

Supporting Information

Synthesis of a New Family of Acyclic Nucleoside Phosphonates, Analogues of TPases Transition States

Bénédicte Dayde,^{a,b} Samira Benzaria,^b Claire Pierra,^b Gilles Gosselin,^{b,c}
Dominique Surleraux,^b Jean-Noel Volle,^a Jean-Luc Pirat,^a David Virieux^{a,*}

^a AM2N, Institut Charles Gerhardt, UMR 5253, ENSCM, 8, rue de l'Ecole Normale, F-34296 Montpellier – France. Fax: +33 (0)467 14 43 19; Tel: +33 (0)467 14 43 14; E-mail: david.virieux@enscm.fr

^b Idenix Pharmaceuticals, Medicinal Chemistry Laboratory, Cap Gamma, 1682 rue de la Valsière, BP50001, 34189 Montpellier Cedex 4 – France.

^c UMR5247 CNRS-UMI-UM2 (IBMM), Université Montpellier 2, 34095 Montpellier Cedex 5, France.

david.virieux@enscm.fr

Table of contents

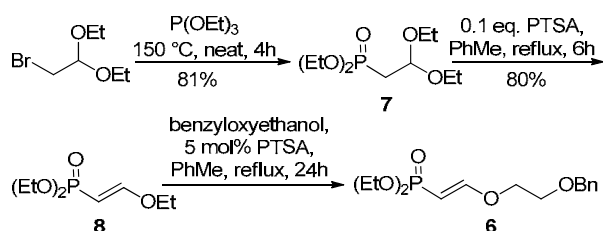
I. General Considerations	2
II. NMR and mass spectra	2
II.1. (E)-diethyl 2-[2-(benzyloxy)ethoxy]vinylphosphonate 6	2
II.2. (±)-(1R,2R)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-1-iodoethylphosphonate 9a	4
II.3. (±)-(1R,2R)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-1-iodoethylphosphonate 9b	10
II.4. (±)-(1R,2R)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]-1-iodoethylphosphonate 9c.....	15
II.5. (+)-Diethyl 2-(2-(benzyloxy)ethoxy)-2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)ethylphosphonate 10a...	21
II.6. (±)-Diethyl 2-(2-(benzyloxy)ethoxy)-2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)ethylphosphonate 10b	25
II.7. (±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate Bz-10c	31
II.8. (±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate 10c	36
II.9. (±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 11a	41
II.10. (±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 11b	47
II.11. (±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 11c	53
II.12. (±)-Lithium ethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 12a	59
II.13. (±)-Lithium ethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 12b	62
II.14. (±)-Lithium ethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 12c.....	65
II.15. (±)-2-(2,4-Dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid triethyl ammonium salt 5a	68
II.16. (±)-2-(5-Methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid 5b...	71
II.17. (±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid 5c.....	74

I. General Considerations

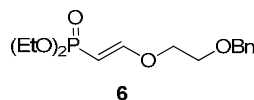
Before use, commercial reagents were purified by distillation or sublimation. All manipulations were carried out using standard Schlenk Techniques. Solvents were dried according to current methods, distilled and stored under nitrogen atmosphere. All reactions involving air or moisture sensitive reagents or intermediates were carried out under dry nitrogen in flame-dried glassware. Melting points were measured on a Büchi B-540 apparatus and are uncorrected. All new compounds were characterized by ^1H NMR, ^{13}C NMR, ^{31}P NMR using a Bruker DRX 400 MHz NMR spectrometer or a Bruker Avance 250 MHz NMR spectrometer. All NMR experiments performed on phosphorus were indicated uncoupling of hydrogen. All NMR led during reaction were done with a closed capillary DMSO-D6 probe in the NMR tube. Low and high resolution mass spectra were determined on an electrospray ionization (ESI) WATERS Micromas Q-ToF spectrometer with as internal reference H_3PO_4 (0.1% in water/acetonitrile, 1:1).

II. NMR and mass spectra

II.1. (*E*)-diethyl 2-[2-(benzyloxy)ethoxy]vinylphosphonate **6**

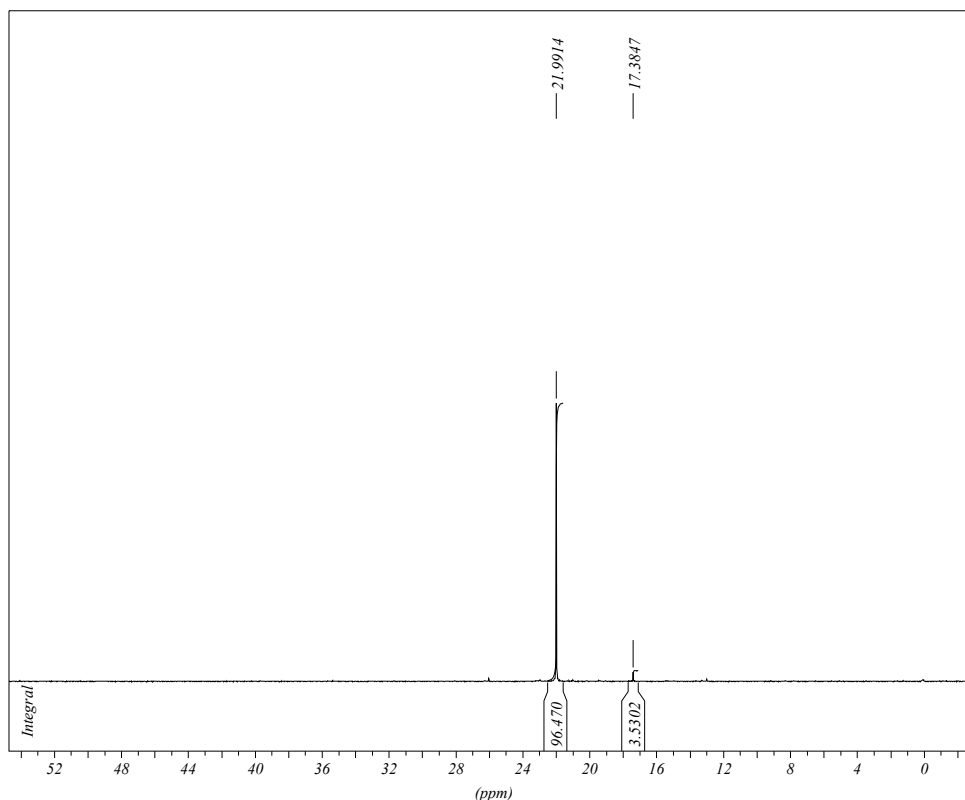


Scheme 1 Synthesis of benzyloxyethoxyethylenylphosphonate **6**



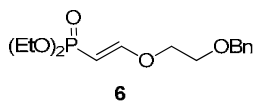
$\text{C}_{15}\text{H}_{23}\text{O}_5\text{P}$
MW = 314.32 g.mol $^{-1}$
Yellow oil (9.09 g, 79%)

bsd02-123 D1 Bouilleur
31P CPD CDC13



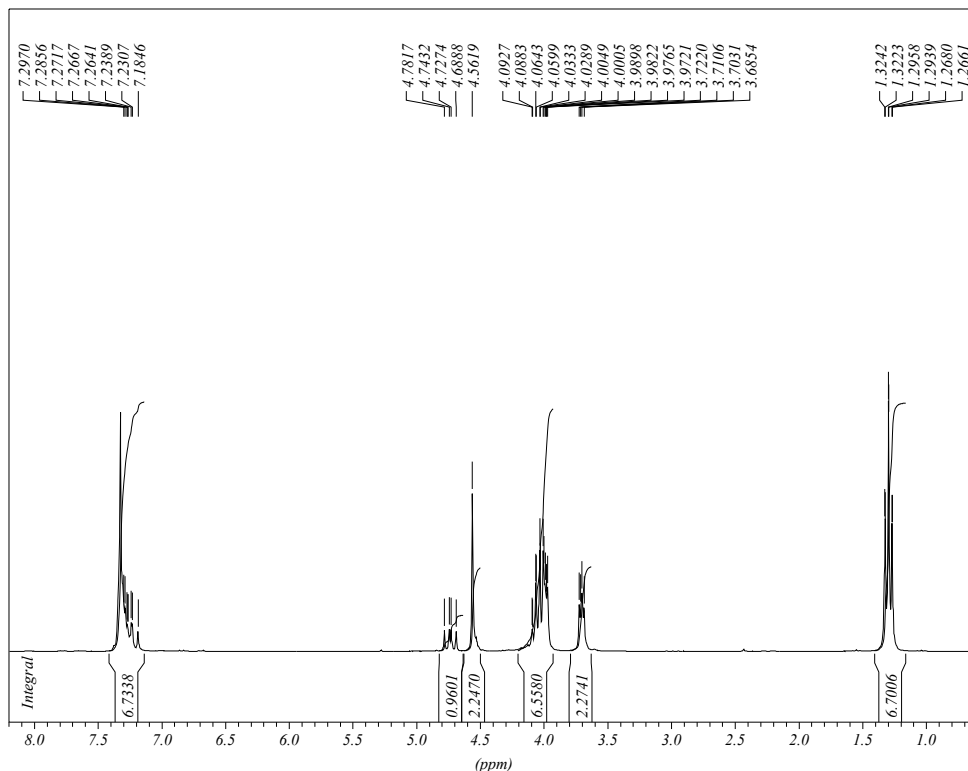
*** Current Data Parameters ***
NAME : BOULL-1
EXPNO : 230
PROCNO : 0
*** Acquisition Parameters ***
DATE_1 : 07:17:14
DATE_d : Feb 11 2009
NS : 16
O1 : -5062.76 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD 2
RG : 10321.2998047
SFO1 : 101.2494172 MHz
SOLVENT : CDCl_3
SW : 401.4878 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 54.73 ppm
Stop : -3.25 ppm
SR : 0.00 Hz
SOLVENT : ?

(E)-diethyl 2-[2-(benzyloxy)ethoxy]vinylphosphonate 6



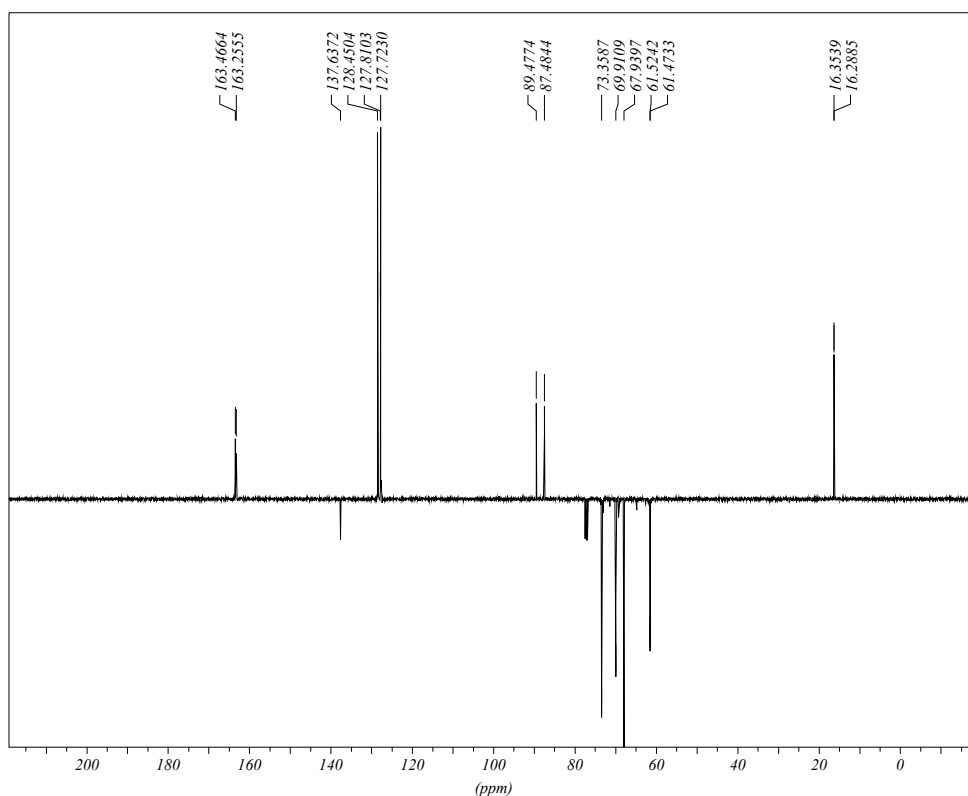
C₁₅H₂₃O₅P
 MW = 314.32 g.mol⁻¹
 Yellow oil (9.09 g, 79%, instable to storage)

bsd02-123 D1 Bouilleur
 1H CDCl₃



*** Current Data Parameters ***
 NAME : BOUILL-1
 EXPNO : 231
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 07:14:26
 DATE_d : Feb 11 2009
 NS : 16
 O1 : 1544.66 Hz
 PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD 2
 RG : 128.000000
 SFO1 : 250.1315447 MHz
 SOLVENT : CDCl₃
 SW : 20.6930 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 8.20 ppm
 Stop : 0.59 ppm
 SR : -0.00 Hz
 SOLVENT : ?

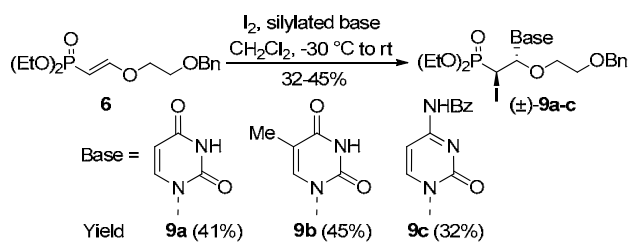
BSD 02 123 bouilleurC13APT CDCl₃ opt/topspin cristau 19



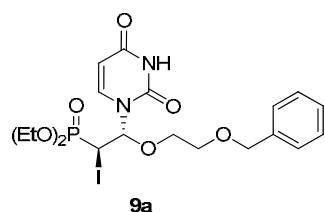
*** Current Data Parameters ***
 NAME : BOUILL-1
 EXPNO : 235
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 05:23:22
 DATE_d : Feb 19 2009
 NS : 256
 O1 : 10060.80 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
 RG : 16384.000000
 SFO1 : 100.6228298 MHz
 SOLVENT : CDCl₃
 SW : 238.3238 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

II.2. (±)-(1*R*,2*R*)-Diethyl iodoethylphosphonate **9a**

2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-

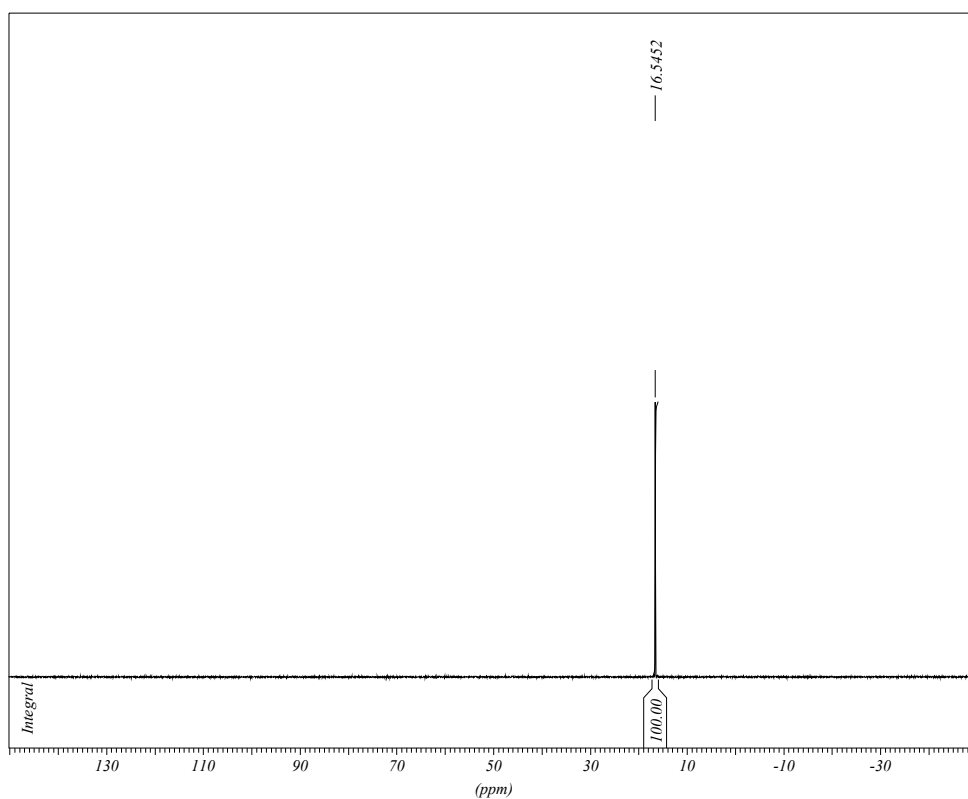


Scheme 2 Iodo-base introduction on **6**



BSD 02 085 F2P31CPD CDC13 opt/topspin cristau 41

$\text{C}_{19}\text{H}_{26}\text{IN}_2\text{O}_7\text{P}$
MW = 552.31 g.mol⁻¹
Colorless oil (0.35 g, 40 %)



*** Current Data Parameters ***

NAME : final
EXPNO : 201
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03-08-32
DATE_d : Jan 06 2009
NS : 16
O1 : 8098.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 20642.5000000
SFO1 : 161.9836718 MHz
SOLVENT : CDCl3

SW : 200.4371 ppm

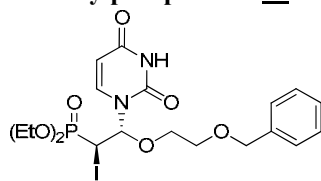
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 150.22 ppm
Stop : -50.22 ppm
SR : 0.01 Hz
SOLVENT : ?

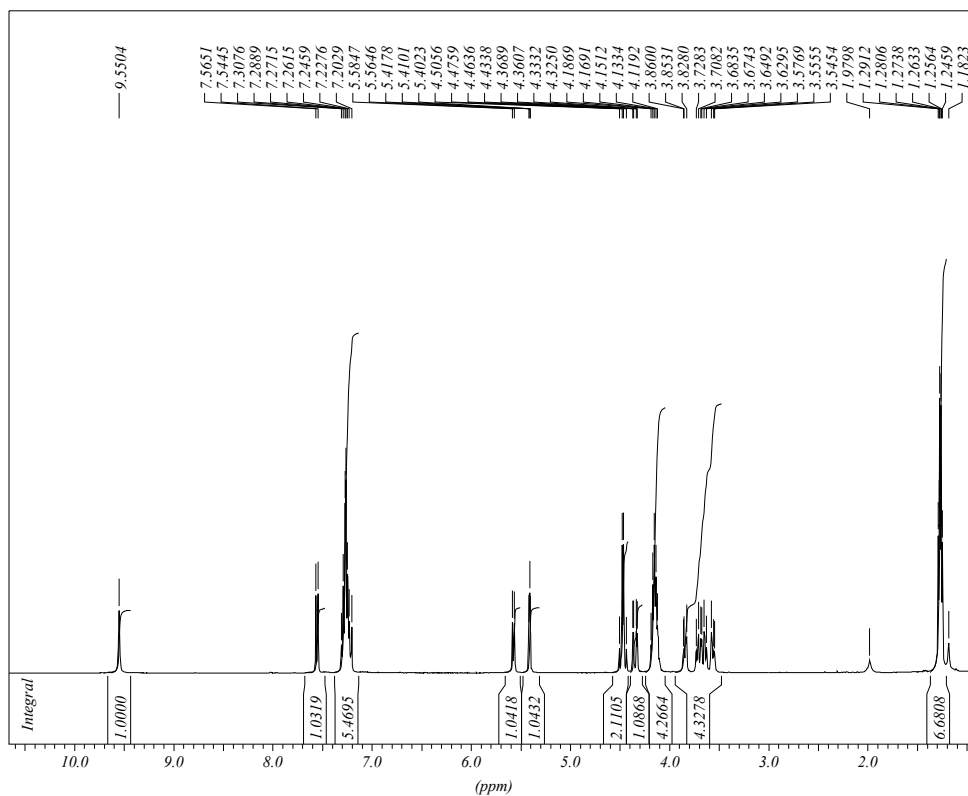
(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-iodoethylphosphonate **9a**



9a

BSD 02 085 F2PROTON CDCl3 opt/topspin cristau 41

C₁₉H₂₆IN₂O₇P
MW = 552.31 g.mol⁻¹
Colorless oil (0.35 g, 40 %)



*** Current Data Parameters ***

NAME : final
EXPNO : 202
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03:11:40
DATE_d : Jan 06 2009
NS : 16
O1 : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 71.8000031
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 14.9831 ppm

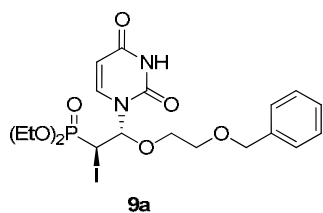
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

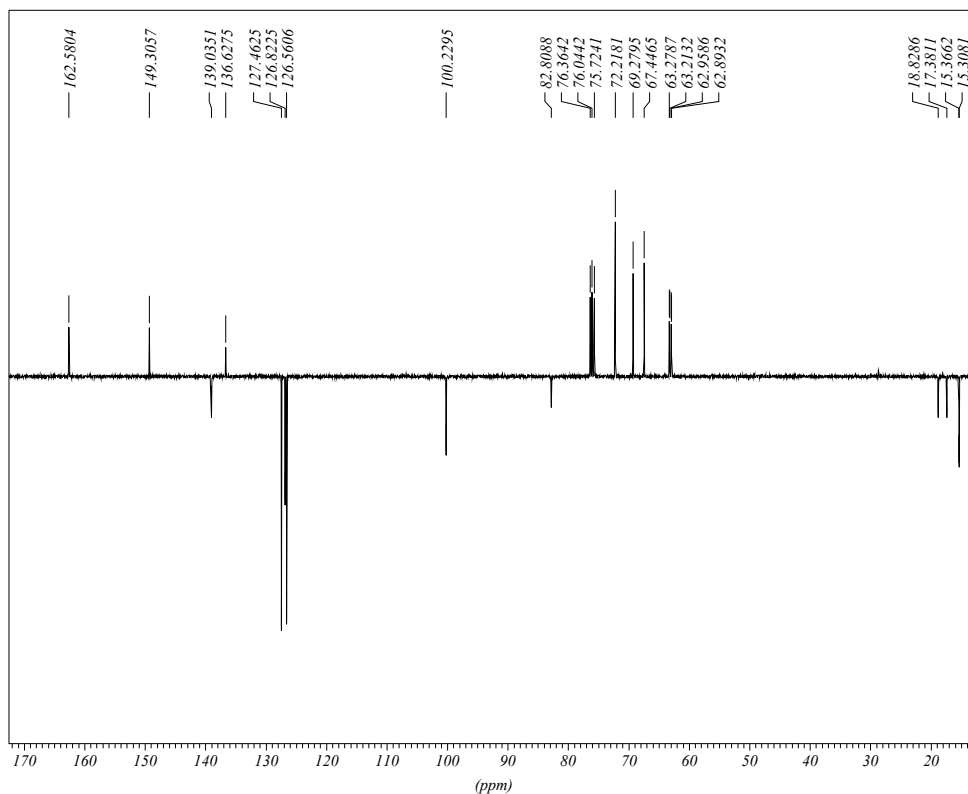
Start : 10.66 ppm
Stop : 0.89 ppm
SR : 32.79 Hz
SOLVENT : ?

(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-iodoethylphosphonate **9a**



C₁₉H₂₆IN₂O₇P
MW = 552.31 g.mol⁻¹
Colorless oil (0.35 g, 40 %)

BSD 02 085 F2C13APT CDC13 opt/topspin cristau 5



*** Current Data Parameters ***

NAME : final
EXPNO : 204
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 14:19:40
DATE_d : Jan 06 2009
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840
RG : 16384.000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm

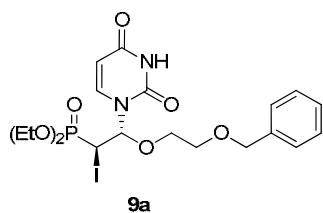
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 172.55 ppm
Stop : 12.17 ppm
SR : 105.23 Hz
SOLVENT : ?

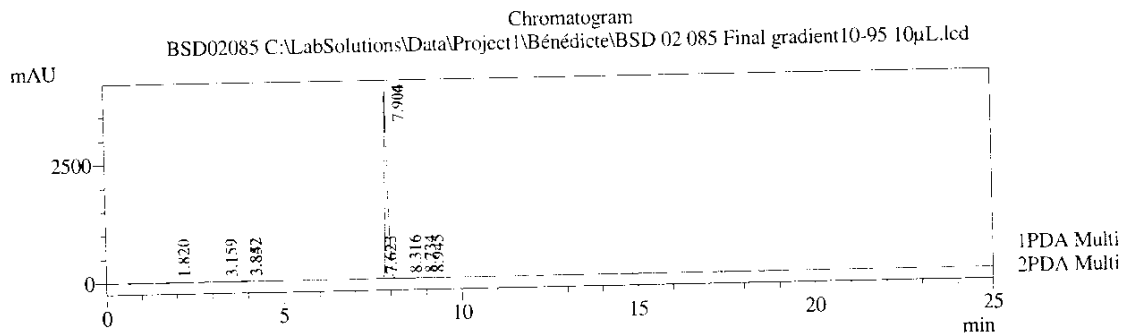
(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-iodoethylphosphonate **9a**



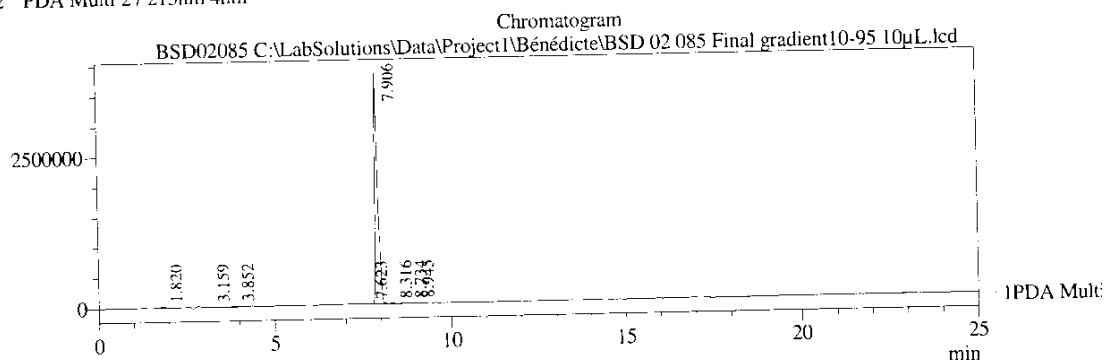
C₁₉H₂₆IN₂O₇P
 MW = 552.31 g.mol⁻¹
 Colorless oil (0.35 g, 40 %)

==== Shimadzu LCsolution Analysis Report ====

C:\...BSD 02 085 Final gradient10-95 10µL.lcd
 Acquired by : Admin
 Sample Name : BSD02085
 Sample ID : BSD02085
 Tray# : 1
 Vial # : 31
 Injection Volume : 10 µL
 Data File Name : BSD 02 085 Final gradie
 Method File Name : Methode Pierre 2.lcm
 Batch File Name : 06-01-09 grad10-95-2.lc
 Report File Name : bene report.lcr
 Data Acquired : 06/01/2009 17:27:06
 Data Processed : 06/01/2009 18:29:01



- 1 PDA Multi 1 / 254nm 4nm
- 2 PDA Multi 2 / 213nm 4nm

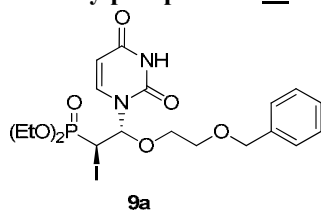


- 1 PDA Multi 2 / 213nm 4nm

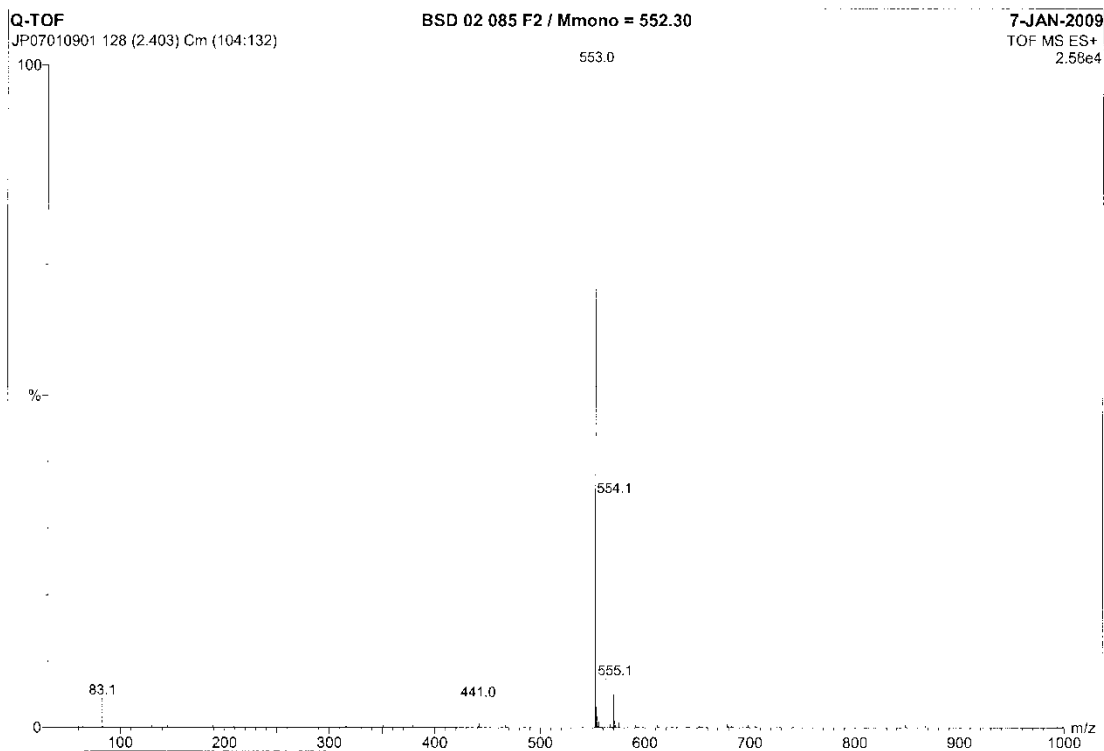
PeakTable

PDA Ch2 213nm 4nm						
Pic	Temps ret.	Hauteur	Aire	Hauteur %	Aire %	
1	1.820	13146	196061	0.337	0.874	
2	3.159	7932	86657	0.204	0.387	
3	3.852	17314	211932	0.444	0.945	
4	7.623	5226	28874	0.134	0.129	
5	7.906	3828054	21705216	98.243	96.808	
6	8.316	8326	68701	0.214	0.306	
7	8.734	5377	56009	0.138	0.250	
8	8.945	11134	67396	0.286	0.301	
Total		3896508	22420846	100.000	100.000	

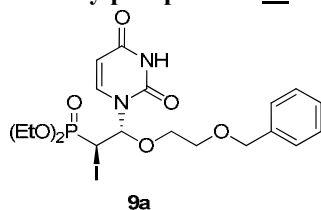
(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-iodoethylphosphonate **9a**



C₁₉H₂₆IN₂O₇P
MW = 552.31 g.mol⁻¹
Colorless oil (0.35 g, 40 %)



(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(2,4-dioxo-3,4-dihydropyrimidin-1(2*H*)-yl)-1-iodoethylphosphonate **9a**



C₁₉H₂₆IN₂O₇P
MW = 552.31 g.mol⁻¹
Colorless oil (0.35 g, 40 %)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

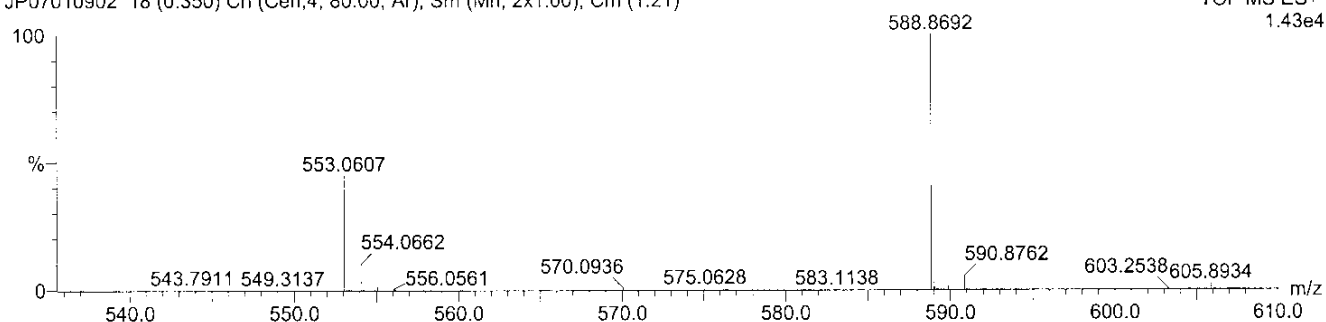
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

50 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 02 085 F2 / Mmono = 552.05
JP07010902 18 (0.350) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (1:21)

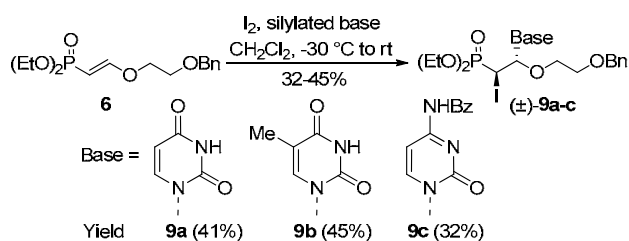
7-JAN-2009
TOF MS ES+
1.43e4



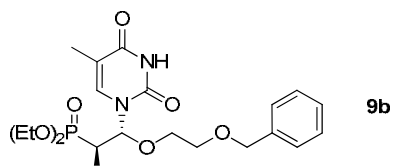
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
553.0607	553.0601	0.6	1.1	7.5	1	C19 H27 N2 O7 P I
	553.0587	2.0	3.6	8.0	2	C17 H25 N5 O6 P I

II.3. (±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-1-iodoethylphosphonate **9b**

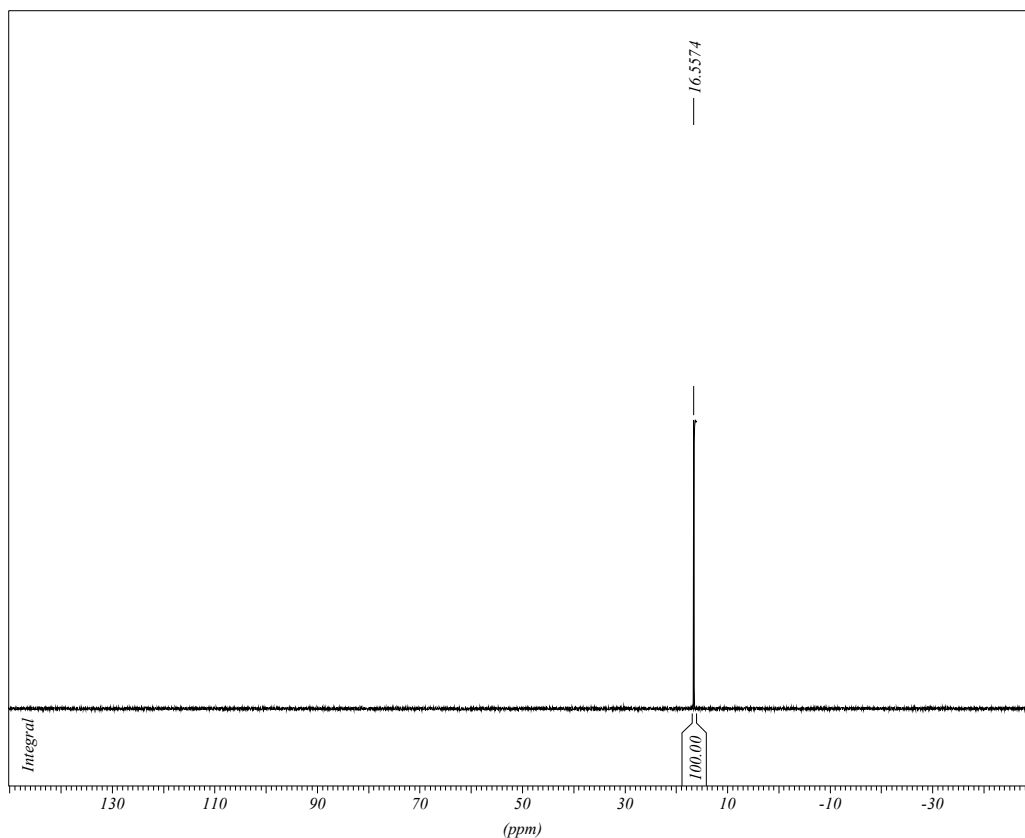


Scheme 2 Iodo-base introduction on **6**



BSD 02 099 F3P31CPD CDC13 opt/topspin cristau 35

$\text{C}_{20}\text{H}_{28}\text{IN}_2\text{O}_7\text{P}$
 MW = 566.33 g.mol⁻¹
 Colorless oil (0.81 g, 45 %)



*** Current Data Parameters ***

NAME : f3
 EXPNO : 210
 PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 15:41:37
 DATE_d : Jan 20 2009
 DE : 6.0 usec
 DS : 4
 NS : 16
 NUC1 : 31P
 O1 : 8098.78 Hz
 O2 : 1600.52 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840

RG : 20642.5000000
 SFO1 : 161.9836718 MHz
 SW : 200.4371 ppm
 SW_h : 32467.532 Hz
 TD : 65536

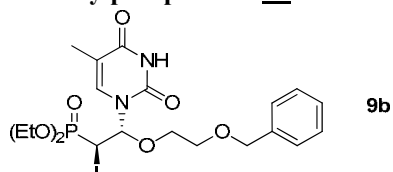
*** Processing Parameters ***

GB : 0.0000000
 LB : 1.00 Hz
 PC : 1.40
 SF : 161.9755730 MHz

*** 1D NMR Plot Parameters ***

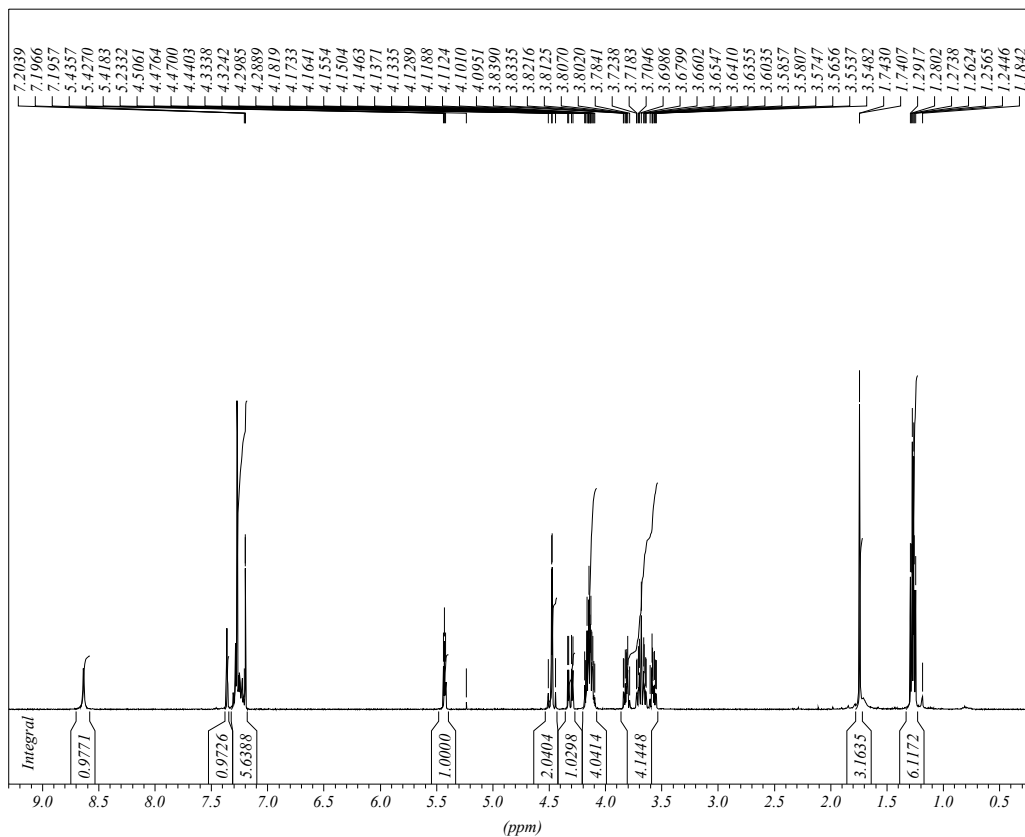
Start : 150.22 ppm
 Stop : -50.22 ppm
 SR : 0.01 Hz
 ppm_cm : 9.46
 Hz_cm : 1531.49

(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2*H*-pyrimidin-1-yl)-1-iodoethylphosphonate **9b**



BSD 02 099 F3PROTON CDCl3 opt/topspin cristau 38

C₂₀H₂₈N₂O₇P
MW = 566.33 g.mol⁻¹
Colorless oil (0.81 g, 45 %)



*** Current Data Parameters ***

NAME : sechage2
EXPNO : 210
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 03:28:26
DATE_d : Jan 27 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G

RG : 143.6999969
SFO1 : 400.1324008 MHz
SW : 14.9831 ppm
SW_h : 5995.204 Hz
TD : 65336

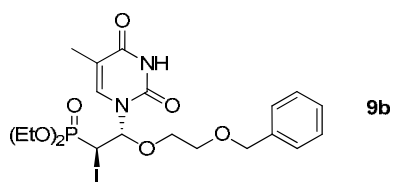
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1300353 MHz

*** 1D NMR Plot Parameters ***

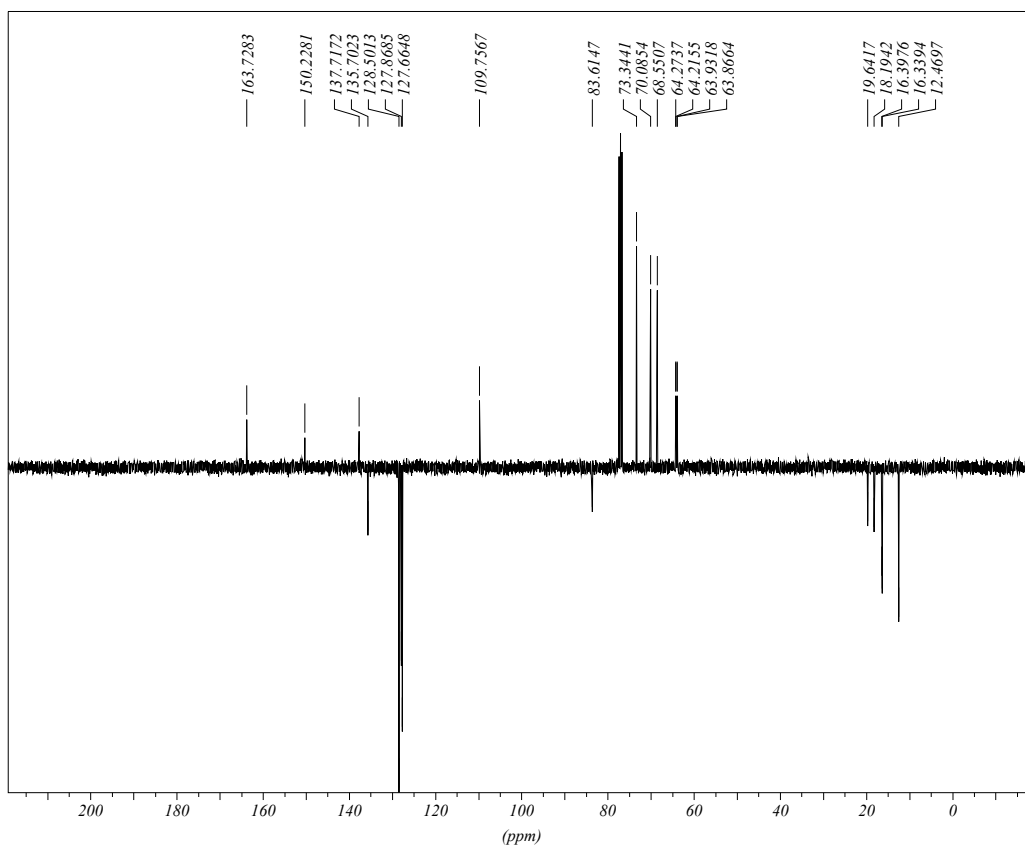
Start : 9.30 ppm
Stop : 0.17 ppm
SR : 35.32 Hz
ppm_cm : 0.43
Hz_cm : 172.27

(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-1-iodoethylphosphonate **9b**



$C_{20}H_{28}IN_2O_7P$
MW = 566.33 g.mol⁻¹
Colorless oil (0.81 g, 45 %)

BSD 02 099 F3C13APT CDC13 opt/topspin cristau 38



*** Current Data Parameters ***

NAME : sechage2
EXPNO : 211
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 16:51:06
DATE_d : Jan 27 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : ¹³C
O1 : 10060.80 Hz
O2 : 1600.52 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840

RG : 23170.500000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

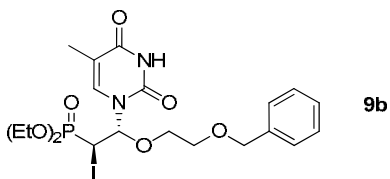
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
ppm_cm : 11.24
Hz_cm : 1131.17

(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2*H*-pyrimidin-1-yl)-1-iodoethylphosphonate **9b**

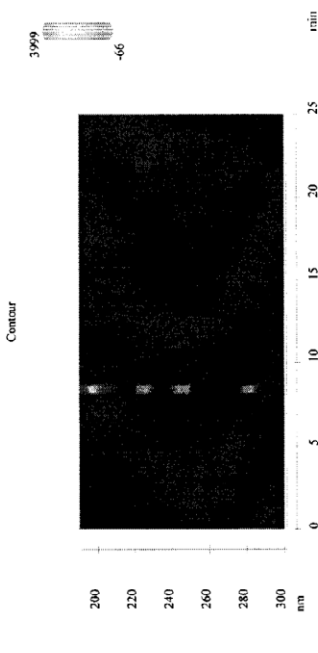
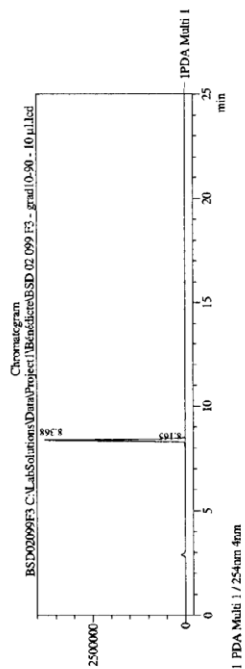


$C_{20}H_{28}IN_2O_7P$
 MW = 566.33 g.mol⁻¹
 Colorless oil (0.81 g, 45 %)

21/01/2009 17:04:45 2 / 2

PDA Ch1 254nm 4nm

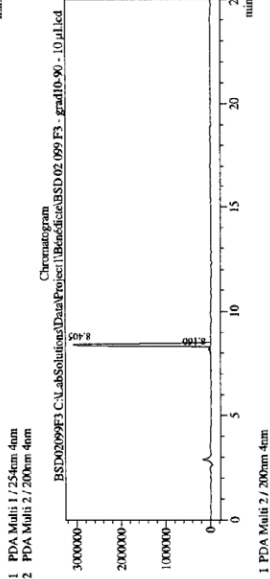
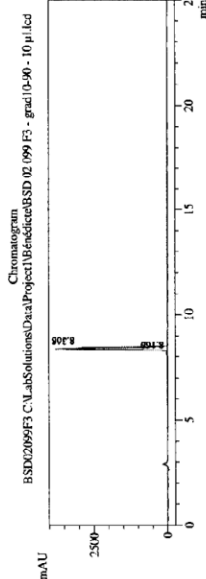
PK	Temps ret.	Hauteur	Aire	% Hauteur	Area %
1	8.165	20613	112779	0.541	0.623
2	8.368	3791862	17920411	99.459	99.377
Total		3812575	18022590	100.000	100.000



21/01/2009 17:04:45 1 / 2

==== Shimadzu LCsolution Analysis Report ====

C:_BSD 02 099 F3 - grad10-90 - 10 µl.lcd
 Acquired by : Admin
 Sample Name : BSD02099F3
 Sample ID : BSD02099F3
 Tray# : 1
 Vial # : 32
 Injection Volume : 10 µL
 Data File Name : BSD 02 099 F3 - grad10
 Backup File Name : BSD02099F3_20090121
 Backup File Name : 21-01-09 grad10-95.tcb
 Report File Name : Detail.lcd
 Date Acquired : 21/01/2009 15:50:20
 Date Processed : 21/01/2009 17:04:25



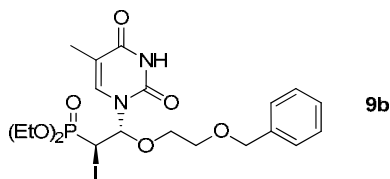
PDA Ch2 200nm 4nm

PK	Temps ret.	Hauteur	Aire	% Hauteur	Area %
1	8.160	42230	346893	1.356	1.295
2	8.405	3084894	25980442	98.654	98.705
Total		3127124	26321335	100.000	100.000

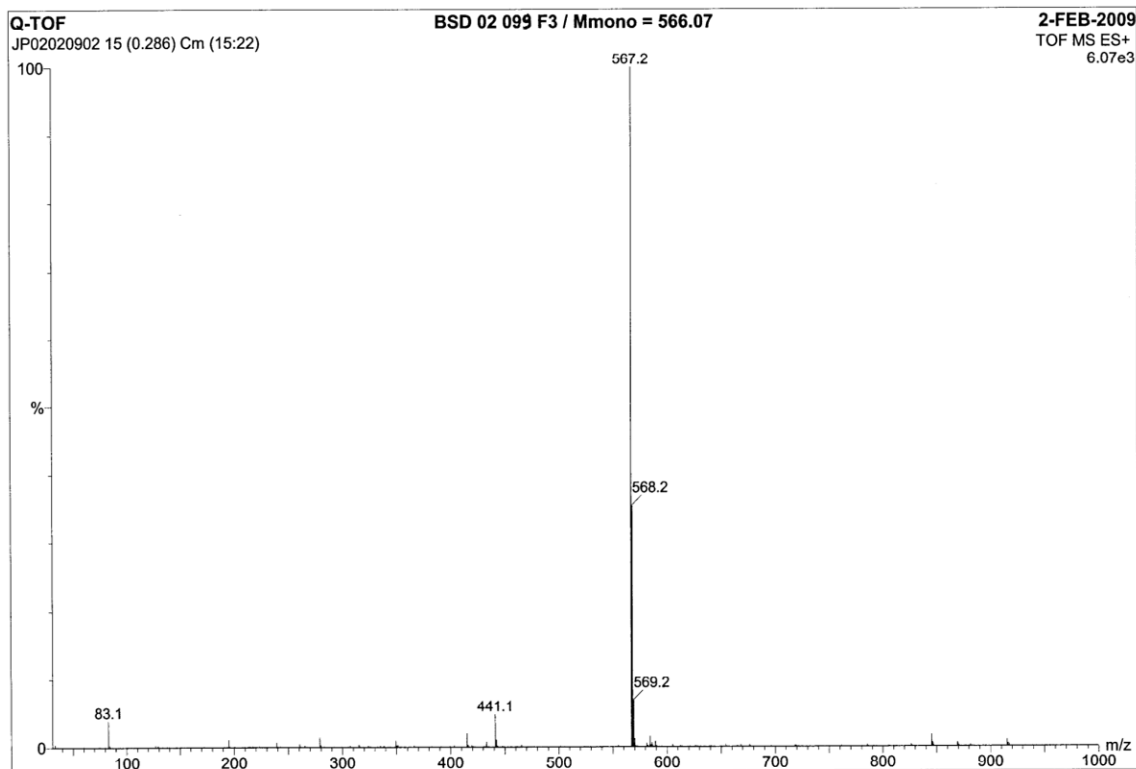
C:\LabSolutions\Data\Project1\Benedicte\BSD 02 099 F3 - grad10-90 - 10 µl.lcd

C:\LabSolutions\Data\Project1\Benedicte\BSD 02 099 F3 - grad10-90 - 10 µl.lcd

(±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-(5-methyl-2,4-dioxo-3,4-dihydro-2*H*-pyrimidin-1-yl)-1-iodoethylphosphonate **9b**



$C_{20}H_{28}IN_2O_7P$
 MW = 566.33 g.mol⁻¹
 Colorless oil (0.81 g, 45 %)



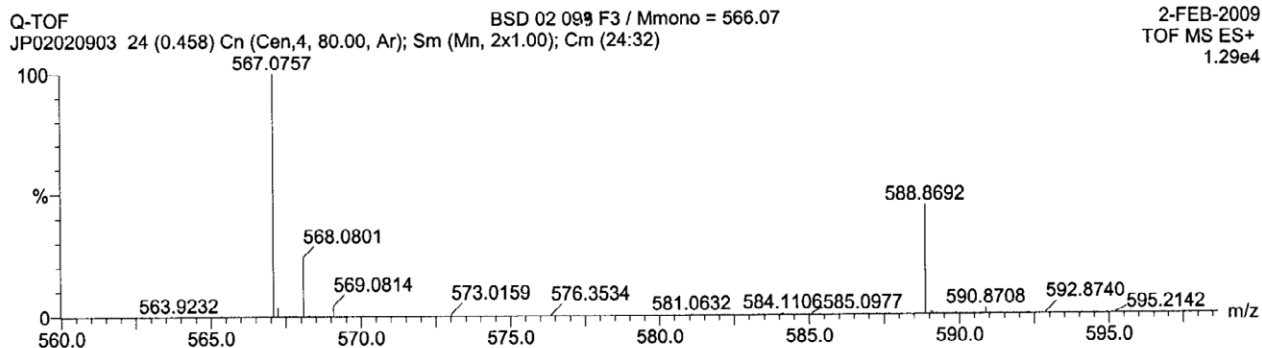
Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 4.0 mDa / DBE: min = -5.0, max = 50.0
 Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

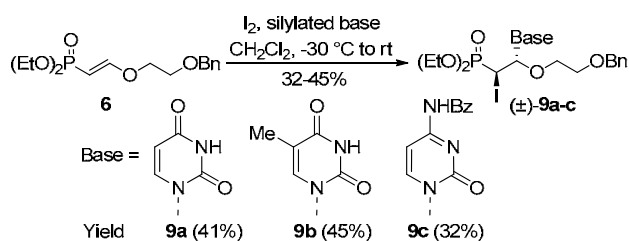
Monoisotopic Mass, Odd and Even Electron Ions
 136 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)



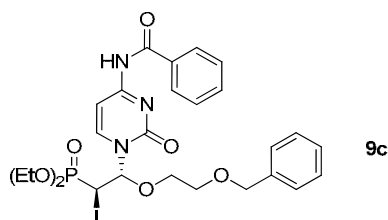
Minimum: -5.0
 Maximum: 4.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
567.0757	567.0757	0.0	0.0	7.5	1	C20 H29 N2 O7 P I
	567.0744	1.3	2.3	8.0	2	C18 H27 N5 O6 P I
	567.0730	2.7	4.7	3.0	3	C17 H31 N O10 P I

II.4. (±)-(1*R*,2*R*)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]-1-iodoethylphosphonate **9c**

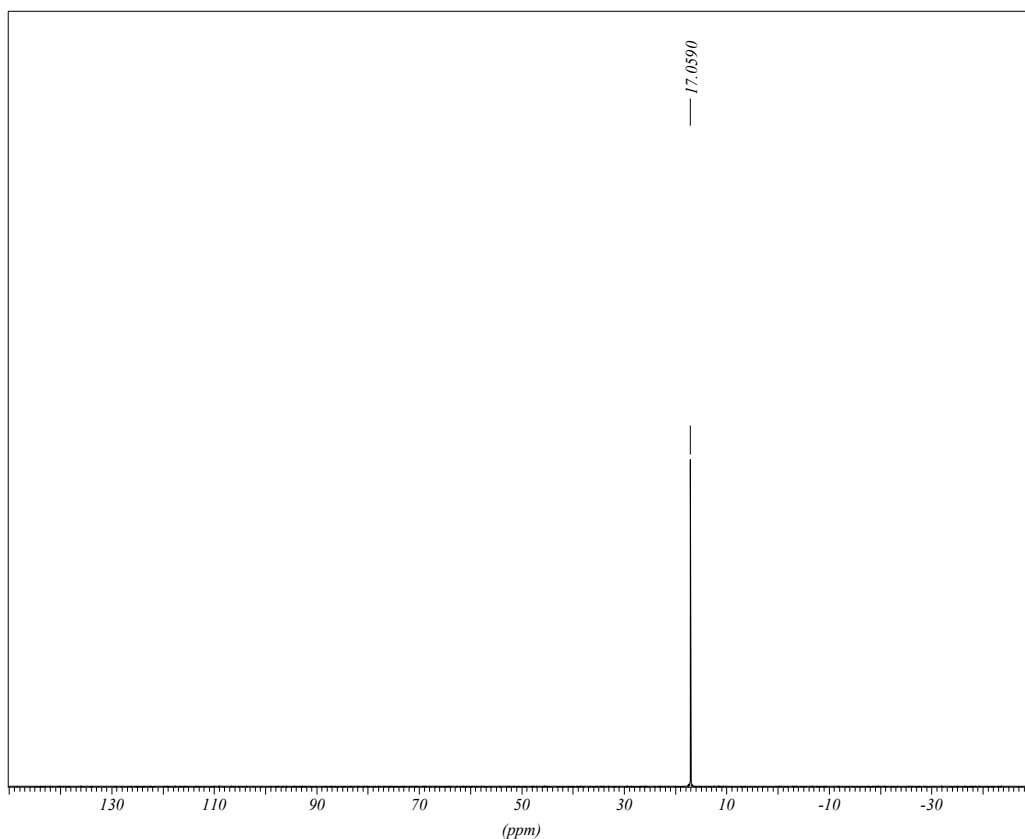


Scheme 2 Iodo-base introduction on **6**



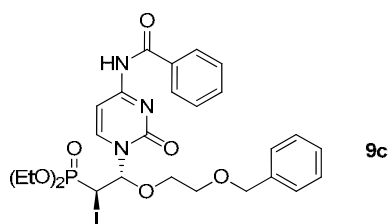
BSD 03 011 F1P31CPD CDC13 opt/topspin cristau 41

$\text{C}_{26}\text{H}_{31}\text{N}_3\text{O}_7\text{IP}$
 MW = 655.43 g.mol⁻¹
 White solid (620 mg, 32 %)



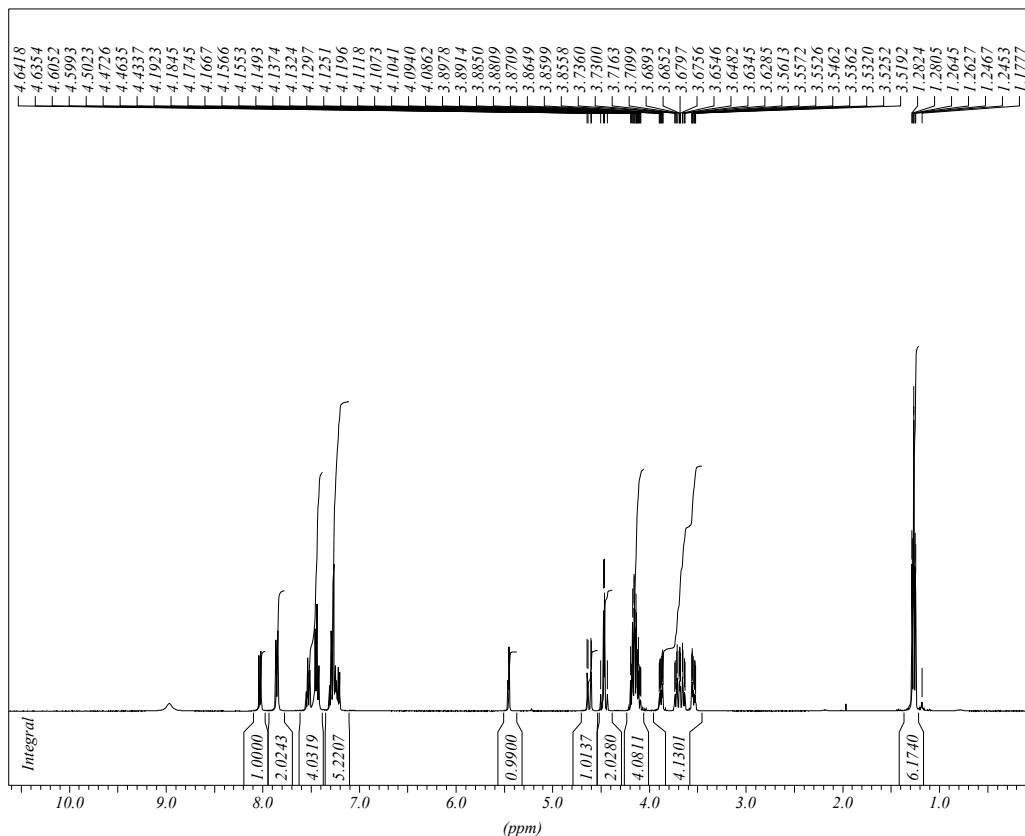
*** Current Data Parameters ***

NAME : f1
 EXPNO : 210
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 23:31:47
 DATE_d : Mar 18 2009
 DE : 6.0 usec
 DS : 4
 NS : 16
 NUC1 : 31P
 O1 : 8098.78 Hz
 O2 : 1600.52 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
 RG : 8192.000000
 SFO1 : 161.9836718 MHz
 SW : 200.4371 ppm
 SW_h : 32467.532 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.000000
 LB : 1.00 Hz
 PC : 1.40
 SF : 161.9755730 MHz
 *** 1D NMR Plot Parameters ***
 Start : 150.22 ppm
 Stop : -50.22 ppm
 SR : 0.01 Hz
 ppm_cm : 9.46
 Hz_cm : 1531.49



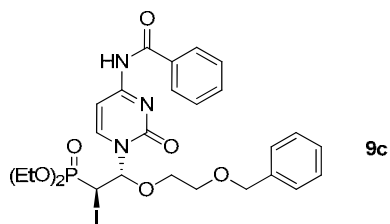
BSD 03 011 F1+F2PROTON CDCl3 opt/topspin cristau 59

$C_{26}H_{31}N_3O_7IP$
 MW = 655.43 g.mol⁻¹
 White solid (620 mg, 32 %)



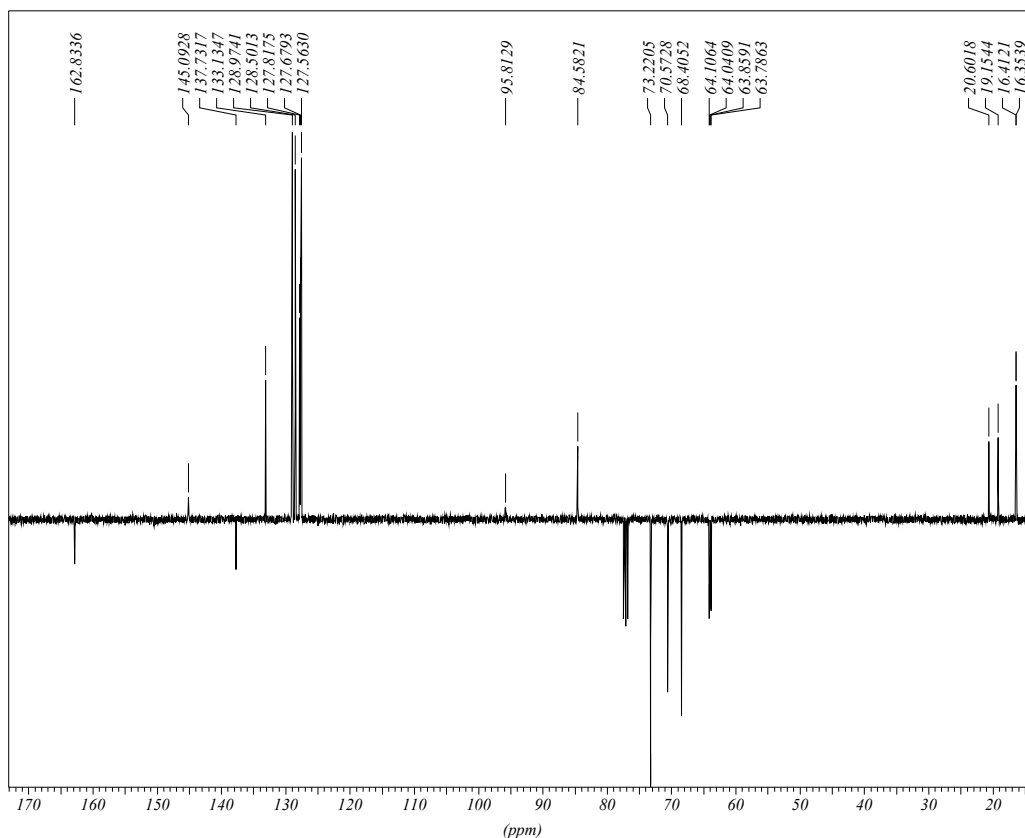
*** Current Data Parameters ***

NAME : pursec
 EXPNO : 202
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 00:32:36
 DATE_d : Mar 20 2009
 DE : 6.0 usec
 DS : 2
 NS : 16
 NUC1 : 1H
 O1 : 2400.78 Hz
 O2 : 2470.97 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840
 RG : 64.0000000
 SFO1 : 400.1324008 MHz
 SW : 14.9831 ppm
 SW_h : 5995.204 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 0.10 Hz
 PC : 1.00
 SF : 400.1300334 MHz
 *** 1D NMR Plot Parameters ***
 Start : 10.63 ppm
 Stop : -0.00 ppm
 SR : 33.37 Hz
 ppm_cm : 0.50
 Hz_cm : 200.69



$C_{26}H_{31}N_3O_7IP$
MW = 655.43 g.mol⁻¹
White solid (620 mg, 32 %)

BSD 03 011 F1+F2C13APT CDC13 opt/topspin cristau 59

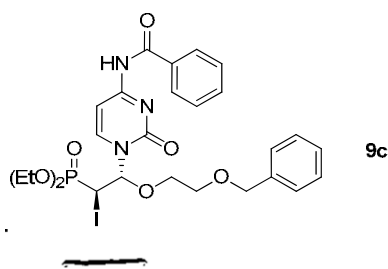


*** Current Data Parameters ***

NAME : pursec
EXPNO : 201
PROCNO : 0
*** Acquisition Parameters ***
DATE_t : 00:29:26
DATE_d : Mar 20 2009
DE : 6.0 usec
DS : 4
NS : 256
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz
*** 1D NMR Plot Parameters ***
Start : 173.11 ppm
Stop : 13.25 ppm
SR : 0.00 Hz
ppm_cm : 7.54
Hz_cm : 758.69

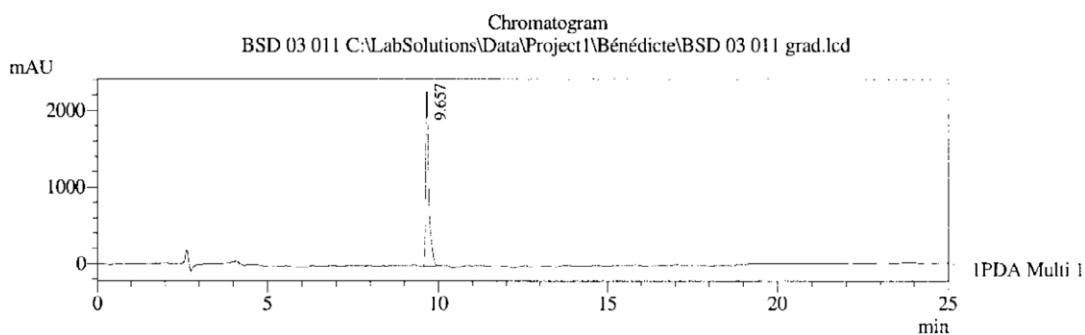


$C_{26}H_{31}N_3O_7IP$
MW = 655.43 g.mol⁻¹
White solid (620 mg, 32 %)

01/04/2009 17:34:17

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Bénédicte\BSD 03 011 grad.lcd
Acquired by : Admin
Sample Name : BSD 03 011
Sample ID : BSD 03 011
Tray# : 1
Vail # : 95
Injection Volume : 10 µL
Data File Name : BSD 03 011 grad.lcd
Method File Name : Methode Pierre 2.lcm
Batch File Name : 01-04-09 grad10-95.lcb
Report File Name : bene report.lcr
Data Acquired : 01/04/2009 17:05:28
Data Processed : 01/04/2009 17:33:48

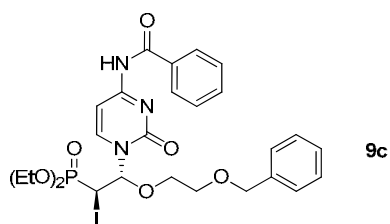


1 PDA Multi 1 / 192nm 4nm

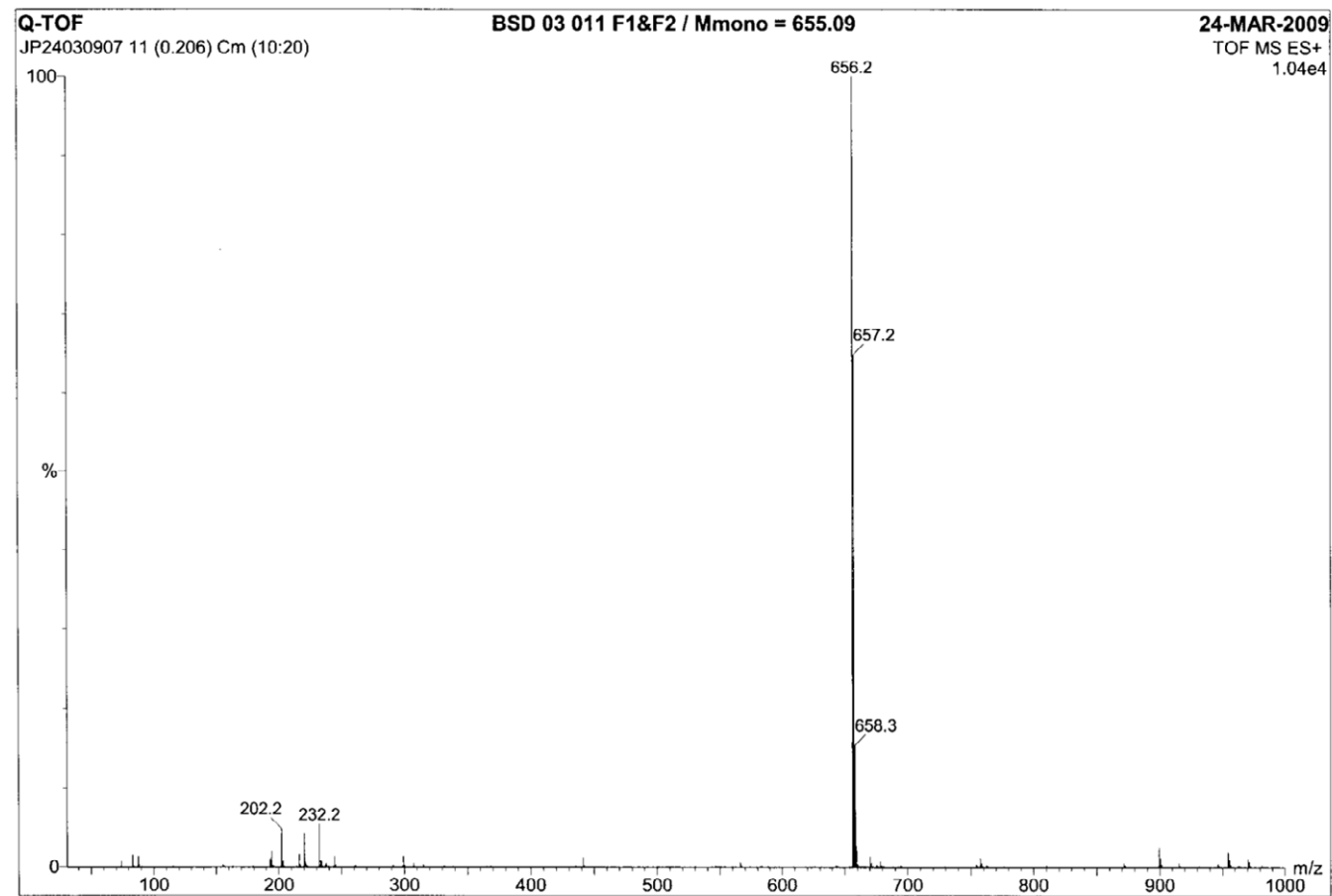
PeakTable

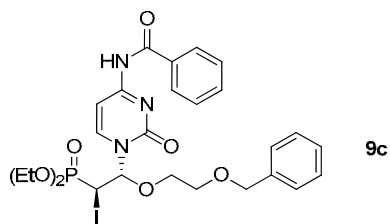
PDA Ch1 192nm 4nm

Pic	Temps rét.	Hauteur	Aire	% Hauteur	Area %
1	9.657	2305109	15658804	100.000	100.000
Total		2305109	15658804	100.000	100.000



$C_{26}H_{31}N_3O_7IP$
MW = 655.43 g.mol⁻¹
White solid (620 mg, 32 %)





$C_{26}H_{31}N_3O_7IP$
MW = 655.43 g.mol⁻¹
White solid (620 mg, 32 %)

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

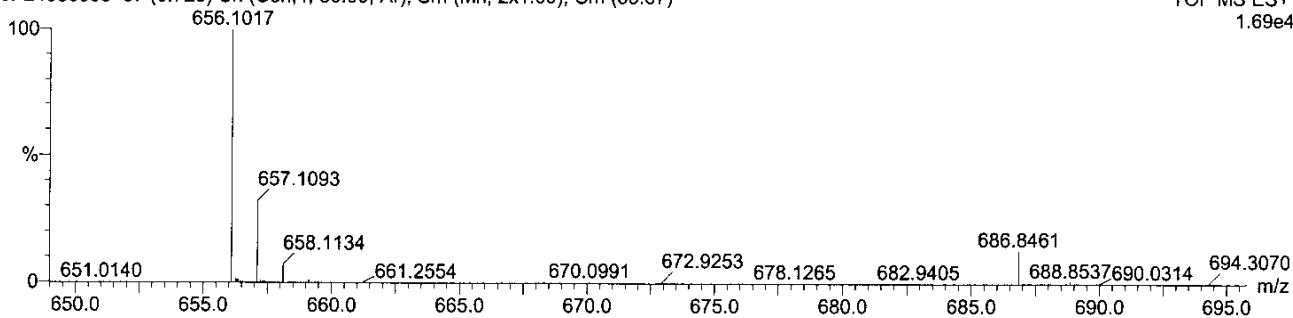
Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

166 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 03 011 F1E2 / Mmono = 655.09
JP24030908 37 (0.728) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (33:37)

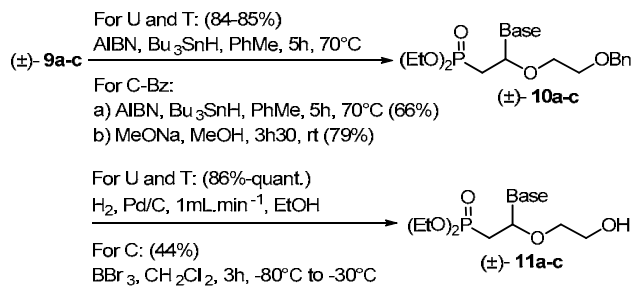
24-MAR-2009
TOF MS ES+
1.69e4



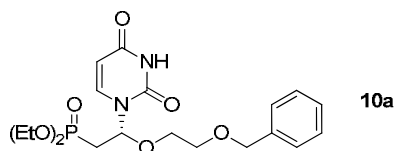
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
656.1017	656.1023	-0.6	-0.9	12.5	1	C26 H32 N3 O7 P I
	656.0996	2.1	3.2	8.0	2	C23 H34 N2 O10 P I

II.5. (±)-Diethyl 2-(2-(benzyloxy)ethoxy)-2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)ethylphosphonate 10a

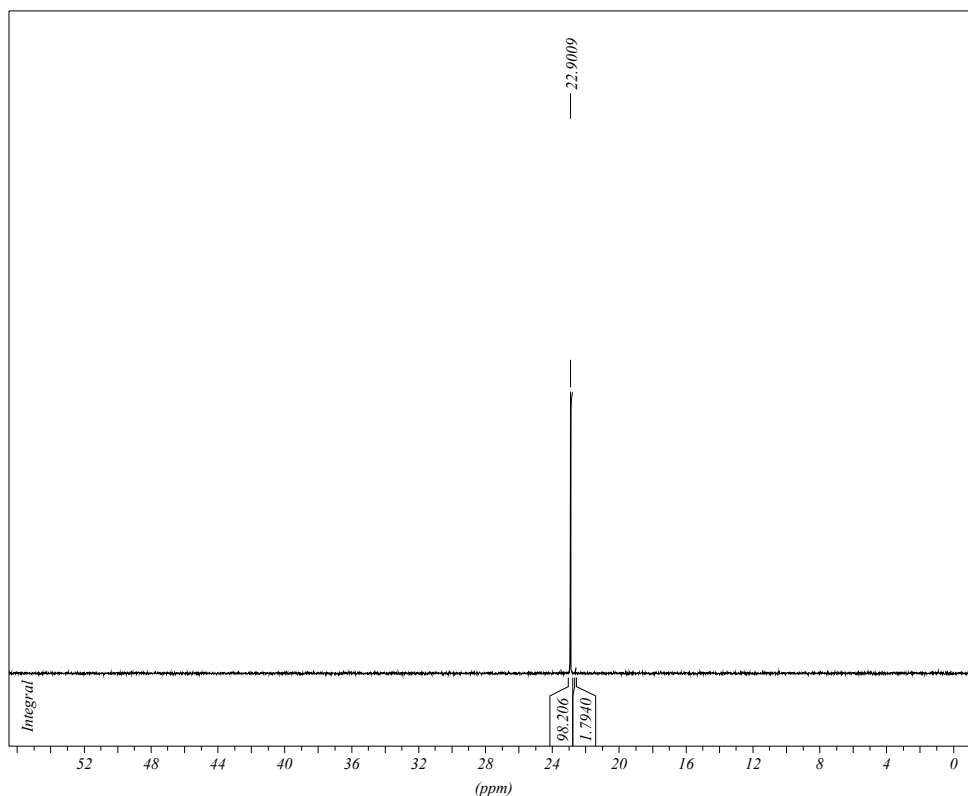


Scheme 3 Reduction and debenzylation steps



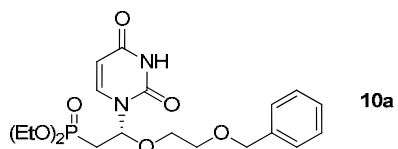
C₁₉H₂₇N₂O₇P
MW = 426.21 g.mol⁻¹
Colorless oil (398 mg, 85 %)

BSD 02 103 F3P31CPD CDC13 opt/topspin cristau 44



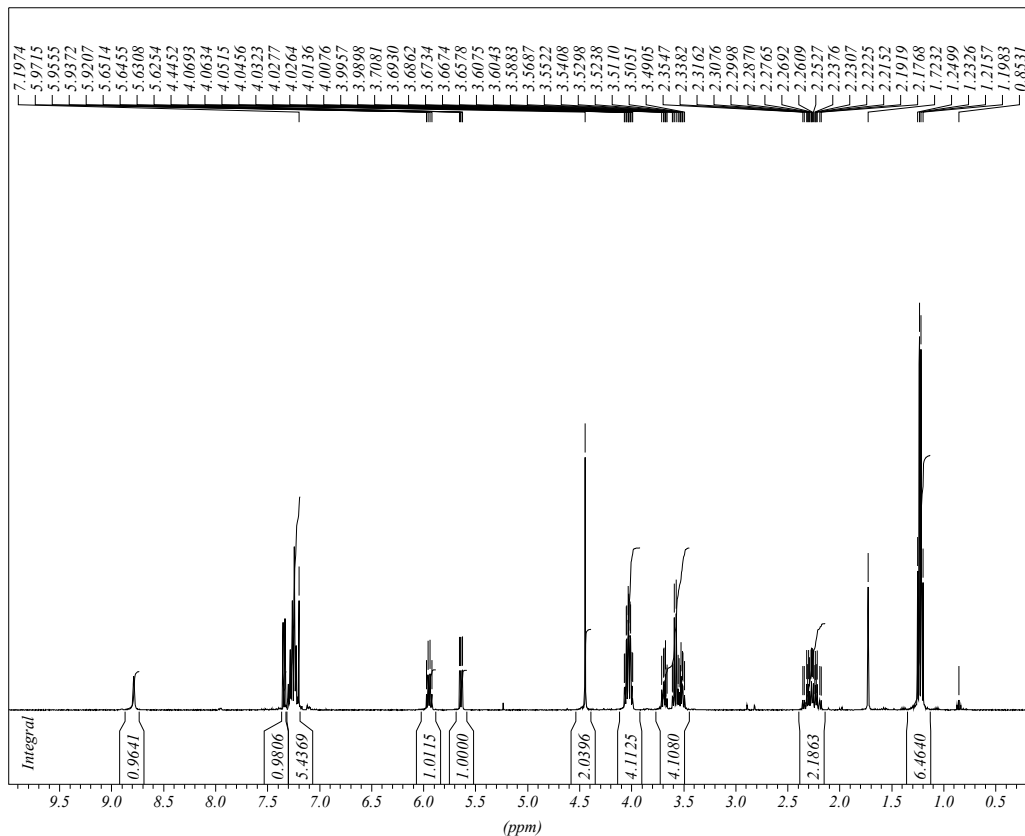
*** Current Data Parameters ***

NAME : F2
EXPNO : 211
PROCNO : 0
*** Acquisition Parameters ***
DATE_1 : 00:10:50
DATE_d : Jan 23 2009
NS : 16
O1 : 8098.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 20642.5000000
SFO1 : 161.9836718 MHz
SOLVENT : CDC13
SW : 200.4371 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 56.50 ppm
Stop : -1.49 ppm
SR : 0.01 Hz
SOLVENT : ?



BSD 02 103 F2PROTON CDCl3 opt/topspin cristau 39

C₁₉H₂₇N₂O₇P
MW = 426.21 g.mol⁻¹
Colorless oil (398 mg, 85 %)



*** Current Data Parameters ***

NAME : sechagel

EXPNO : 212

PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 03:35:55

DATE_d : Jan 27 2009

DE : 6.0 usec

DS : 2

NS : 16

NUC1 : 1H

O1 : 2400.78 Hz

O2 : 2470.97 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD ZS40

RG : 143.6999969

SFO1 : 400.1324008 MHz

SW : 14.9831 ppm

SW_h : 5995.204 Hz

TD : 65536

*** Processing Parameters ***

GB : 0.0000000

LB : 0.10 Hz

PC : 1.00

SF : 400.1300348 MHz

*** 1D NMR Plot Parameters ***

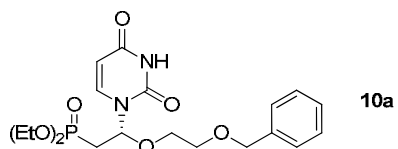
Start : 9.99 ppm

Stop : 0.11 ppm

SR : 34.82 Hz

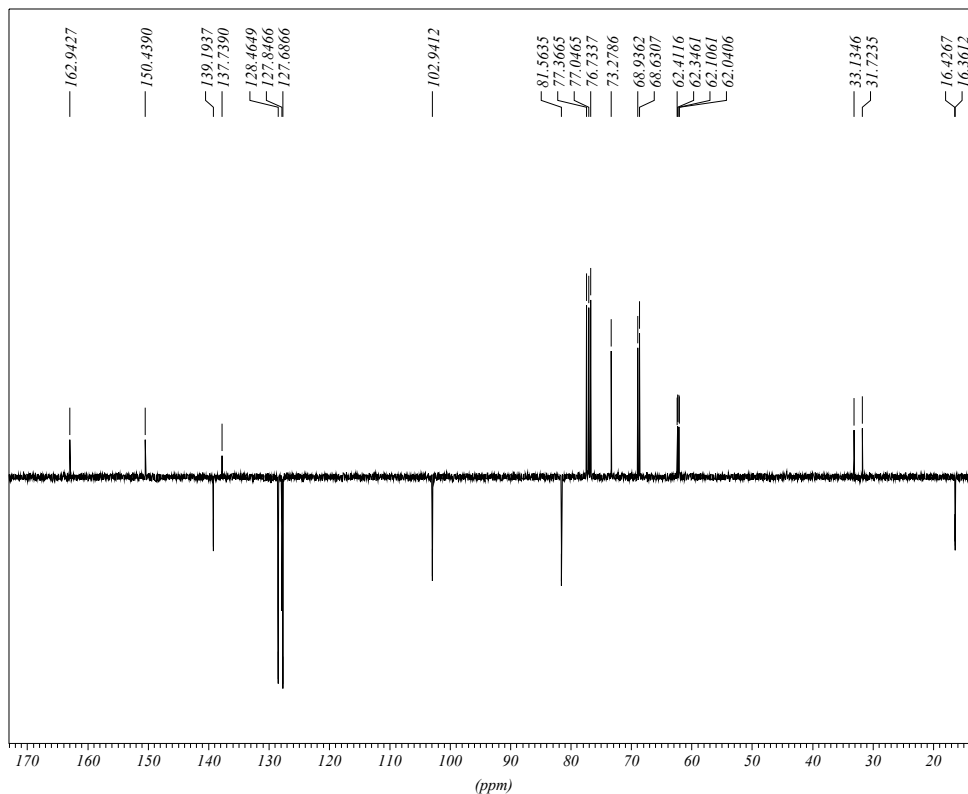
ppm_cm : 0.47

Hz_cm : 186.49

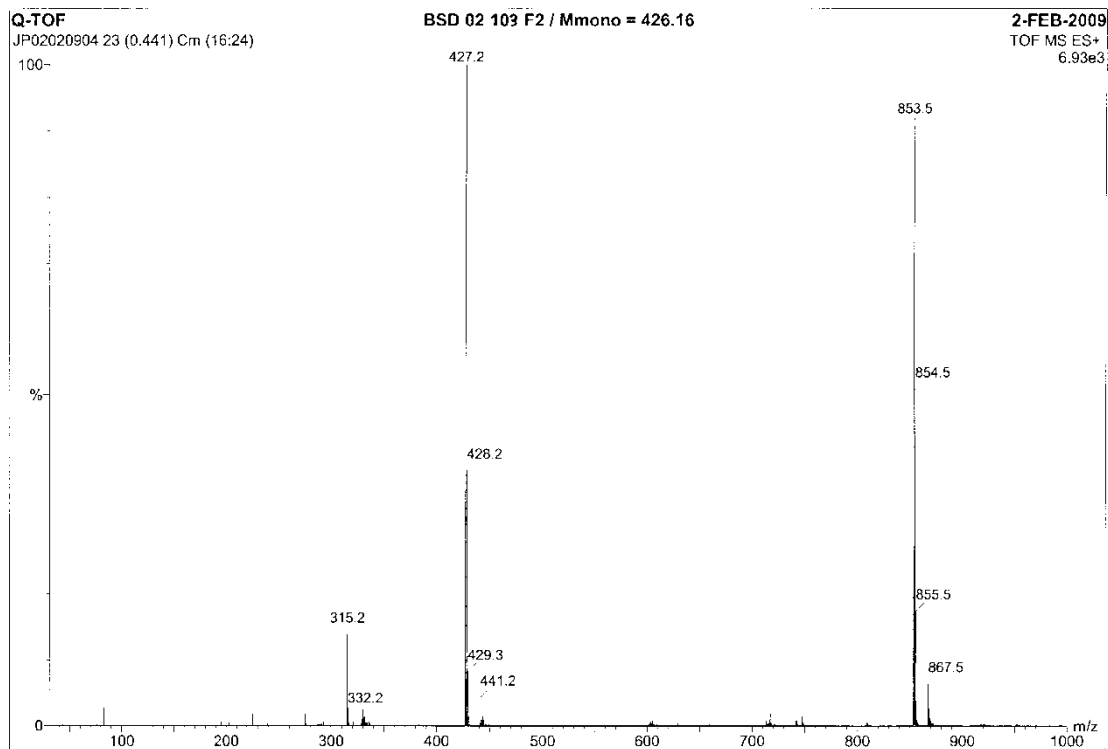


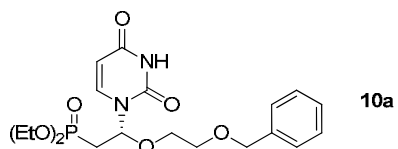
BSD 02 103 F2C13APT CDC13 opt/topspin cristau 39

$C_{19}H_{27}N_2O_7P$
 MW = 426.21 g.mol⁻¹
 Colorless oil (398 mg, 85 %)



*** Current Data Parameters ***
 NAME : sechage1
 EXPNO : 213
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 17:55:34
 DATE_d : Jan 27 2009
 NS : 1024
 O1 : 10060.80 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
 RG : 16384.000000
 SFO1 : 100.6228298 MHz
 SOLVENT : CDC13
 SW : 238.3238 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 173.06 ppm
 Stop : 12.55 ppm
 SR : 0.00 Hz
 SOLVENT : ?





C₁₉H₂₇N₂O₇P
MW = 426.21 g.mol⁻¹
Colorless oil (398 mg, 85 %)

Elemental Composition Report

Page 1

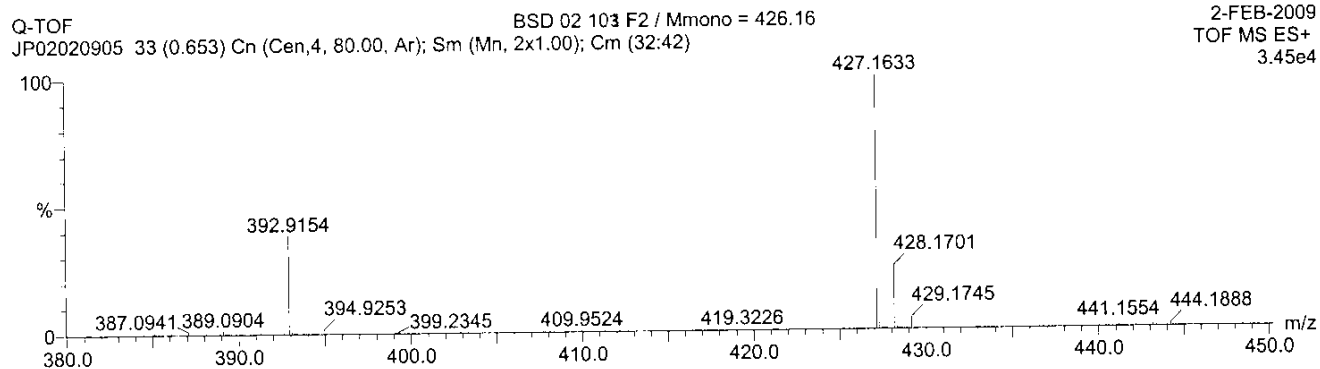
Single Mass Analysis

Tolerance = 4.0 mDa / DBE: min = -5.0, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

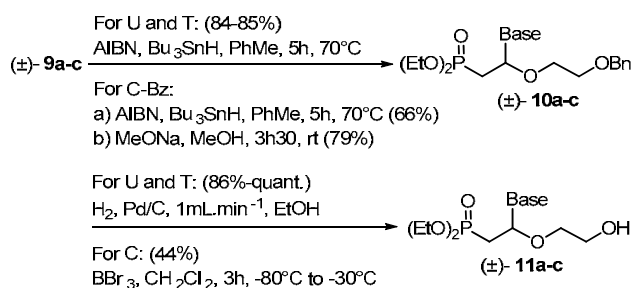
132 formula(e) evaluated with 4 results within limits (up to 50 closest results for each mass)



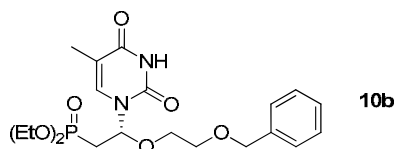
Minimum: -5.0
Maximum: 4.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
427.1633	427.1634	-0.1	-0.3	7.5	1	C19 H28 N2 O7 P
	427.1621	1.2	2.9	8.0	2	C17 H26 N5 O6 P
	427.1607	2.6	6.0	3.0	3	C16 H30 N O10 P
	427.1594	3.9	9.1	3.5	4	C14 H28 N4 O9 P

II.6. (±)-Diethyl 2-(2-(benzyloxy)ethoxy)-2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)ethylphosphonate 10b

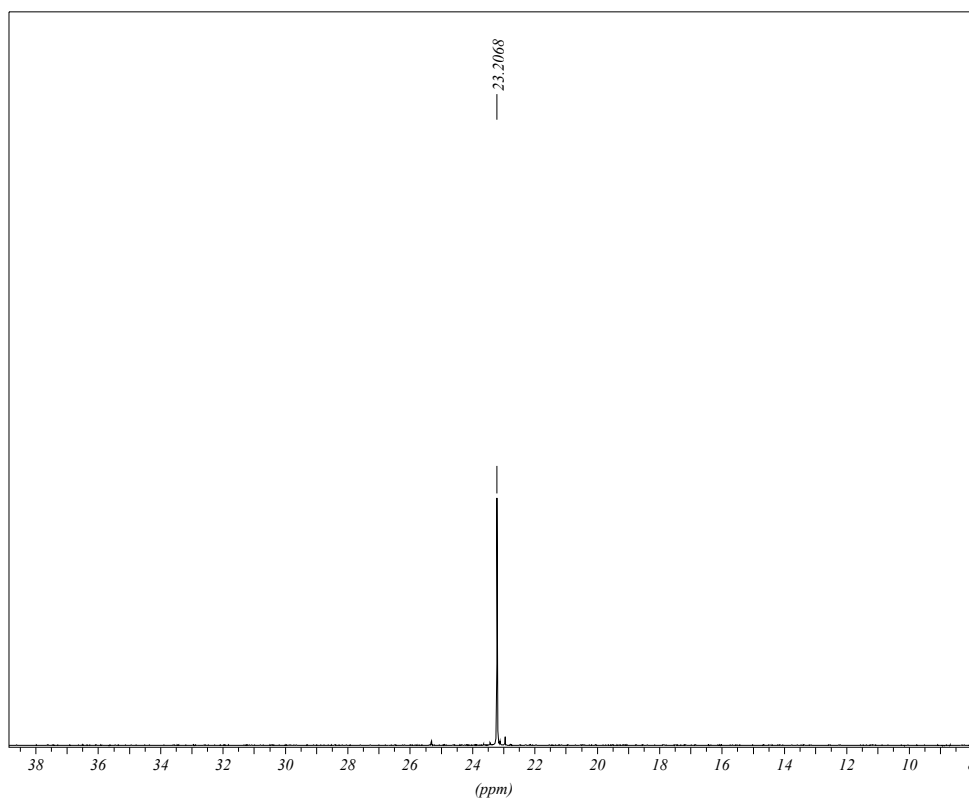


Scheme 3 Reduction and debenzylation steps

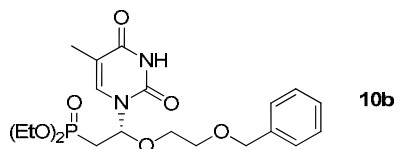


C₂₀H₂₉N₂O₇P
 MW = 440.44 g.mol⁻¹
 Colorless (416 mg, 84 %)

BSD 02 105 F1P3ICPD CDC13 opt/topspin cristau 46

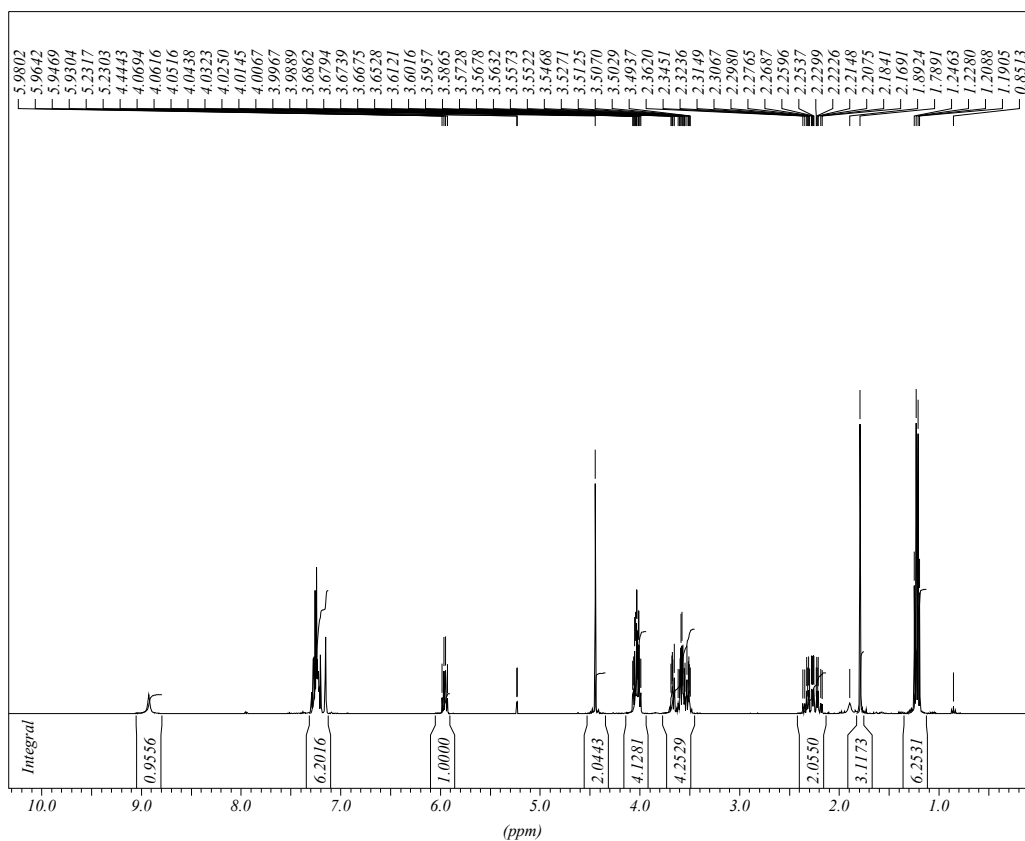


*** Current Data Parameters ***
 NAME : F1
 EXPNO : 214
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 00:28:53
 DATE_d : Jan 23 2009
 NS : 16
 O1 : 8098.78 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
 RG : 13004.0000000
 SFO1 : 161.9836718 MHz
 SOLVENT : CDC13
 SW : 200.4371 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 38.87 ppm
 Stop : 7.77 ppm
 SR : 0.01 Hz
 SOLVENT : ?



$C_{20}H_{29}N_2O_7P$
 MW = 440.44 g.mol⁻¹
 Colorless (416 mg, 84 %)

BSD 02 105 F2PROTON CDC13 opt/topspin cristau 40



*** Current Data Parameters ***

NAME : sechagel
 EXPNO : 214
 PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03:43:38
 DATE_d : Jan 27 2009
 DE : 6.0 usec
 DS : 2
 NS : 16
 NUC1 : 1H
 O1 : 2400.78 Hz
 O2 : 2470.97 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840

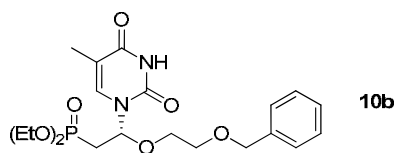
RG : 90.5000000
 SFO1 : 400.1324008 MHz
 SW : 14.9831 ppm
 SW_h : 5995.204 Hz
 TD : 65536

*** Processing Parameters ***

GB : 0.0000000
 LB : 0.10 Hz
 PC : 1.00
 SF : 400.1300332 MHz

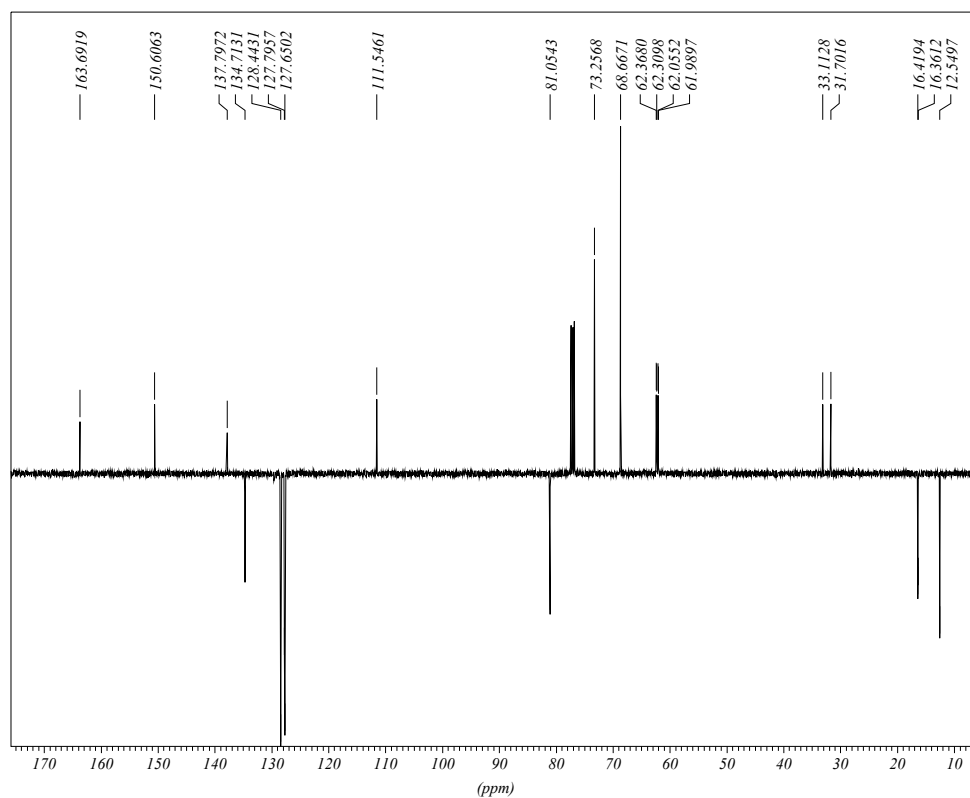
*** 1D NMR Plot Parameters ***

Start : 10.33 ppm
 Stop : 0.02 ppm
 SR : 33.16 Hz
 ppm_cm : 0.49
 Hz_cm : 194.57



$C_{20}H_{29}N_2O_7P$
MW = 440.44 g.mol⁻¹
Colorless (416 mg, 84 %)

BSD 02 105 F2C13APT CDCl3 opt/topspin cristau 40



*** Current Data Parameters ***

NAME : sechage1
EXPNO : 215
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 19:00:03
DATE_d : Jan 27 2009
NS : 1024
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 23170.5000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm

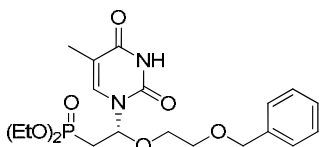
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 175.88 ppm
Stop : 5.42 ppm
SR : 0.00 Hz
SOLVENT : ?

26/01/2009 18:24:28 2 / 2



10b

$C_{20}H_{29}N_2O_7P$
 MW = 440.44 g.mol⁻¹
 Colorless (416 mg, 84 %)

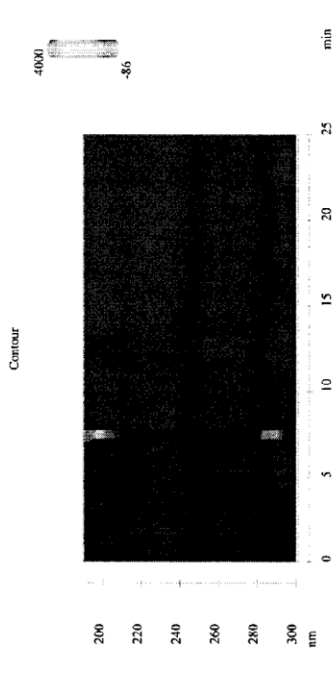
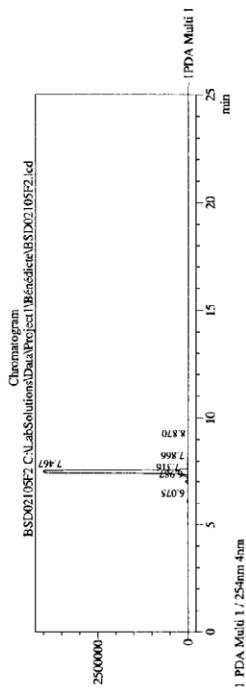
26/01/2009 18:24:28 1 / 2

==== Shimadzu LCsolution Analysis Report ====

Acquired by : C:\LabSolutions\Data\Project1\Bénédicta\BSD02105F2.lcd
 Sample Name : Admin
 Sample ID : BSD02105F2
 Tray# : 1
 Vial # : 92
 Injection Volume : 10 µL
 Data File Name : BSD02105F2.lcd
 Method Name : Methode Pierre 2.lcm
 Batch File Name : 26-01-09 grad10-95.lcb
 Report File Name : Bénédicta report 17-28-43
 Data Acquired : 26/01/2009 17:28:43
 Data Processed : 26/01/2009 18:23:57

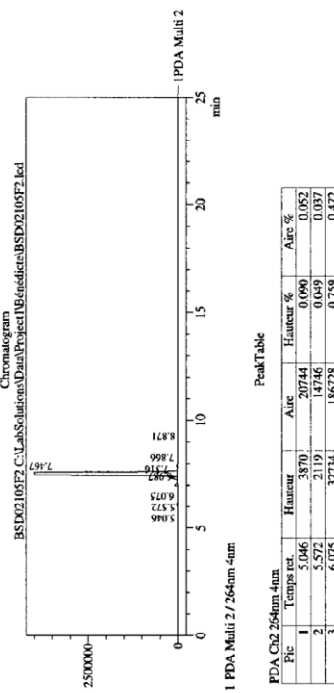
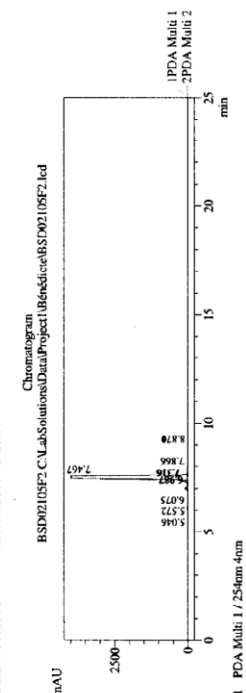
Peak Table

Pic	Temp. ret.	Hauteur	Aire	% Hauteur	Area %
1	6.075	70532	99659	0.484	0.267
2	6.987	70459	393384	1.654	1.050
3	7.316	146735	870762	3.447	2.325
4	7.467	3992856	35894164	93.804	95.943
5	7.866	23387	179409	0.561	0.476
6	8.870	2119	14323	0.050	0.038
Total		4256708	37451001	100.000	100.000



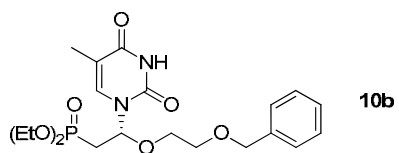
Peak Table

Pic	Temp. ret.	Hauteur	Aire	% Hauteur	Area %
1	3.046	3170	20744	0.090	0.052
2	6.072	37134	185726	0.759	0.477
3	6.987	86735	483186	2.680	1.221
4	7.316	169231	865663	3.922	2.183
5	7.467	3983615	37768268	92.319	95.446
6	7.866	31201	217353	0.723	0.550
7	7.866	2548	15259	0.059	0.039
8	8.871	4315062	39570127	100.000	100.000
Total					

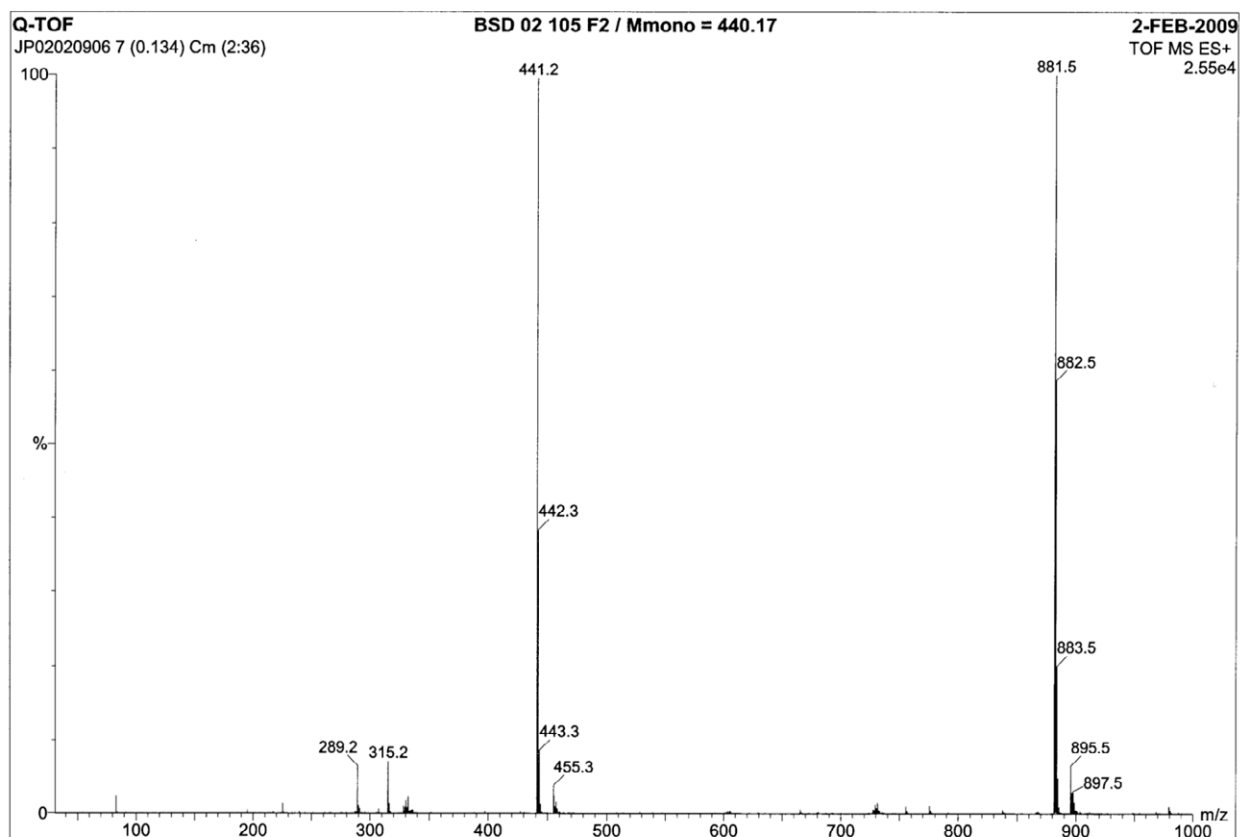


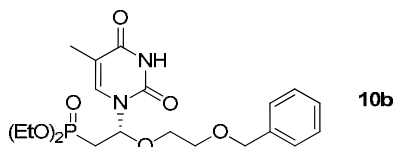
C:\LabSolutions\Data\Project1\Bénédicta\BSD02105F2.lcd

C:\LabSolutions\Data\Project1\Bénédicta\BSD02105F2.lcd



$C_{20}H_{29}N_2O_7P$
MW = 440.44 g.mol⁻¹
Colorless (416 mg, 84 %)





$C_{20}H_{29}N_2O_7P$
MW = 440.44 g.mol⁻¹
Colorless (416 mg, 84 %)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 4.0 mDa / DBE: min = -5.0, max = 50.0

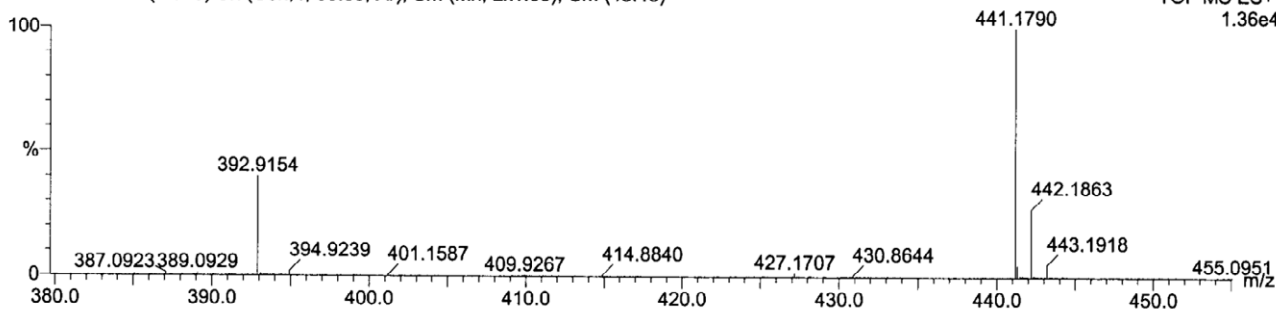
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

136 formula(e) evaluated with 4 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 02 105 F2 / Mmono = 440.17
JP02020907 45 (0.846) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (43:46)

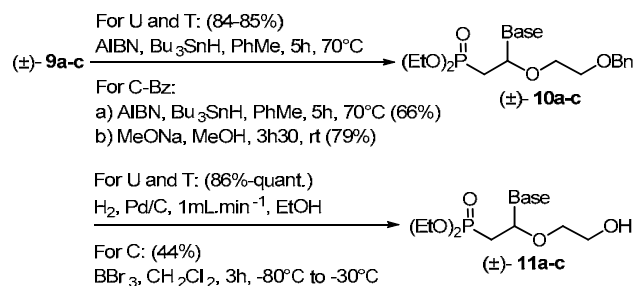
2-FEB-2009
TOF MS ES+
1.36e4



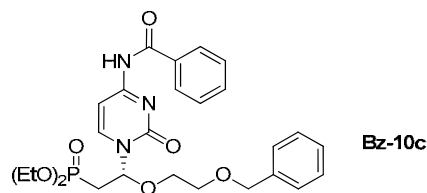
Minimum: -5.0
Maximum: 4.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
441.1790	441.1791	-0.1	-0.1	7.5	1	C20 H30 N2 O7 P
	441.1777	1.3	2.9	8.0	2	C18 H28 N5 O6 P
	441.1764	2.6	5.9	3.0	3	C17 H32 N O10 P
	441.1750	4.0	9.0	3.5	4	C15 H30 N4 O9 P

II.7. (±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate **Bz-10c**

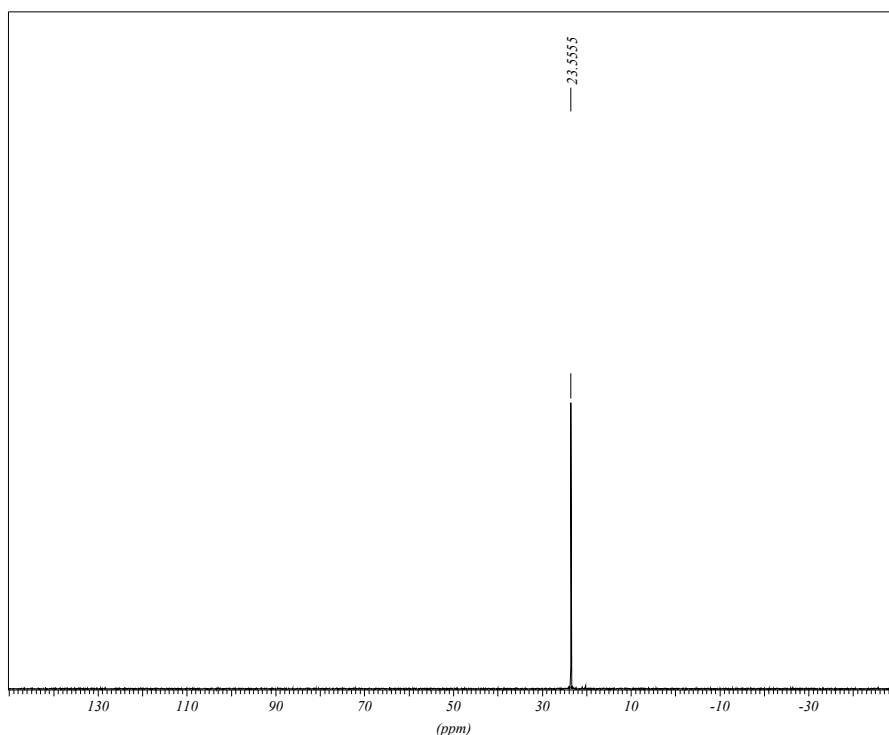


Scheme 3 Reduction and debenzylation steps



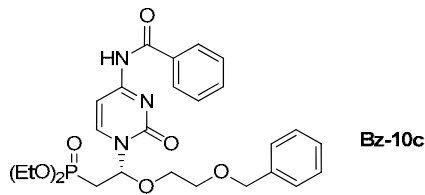
C₂₆H₃₂N₃O₇P
 MW = 529.53 g.mol⁻¹
 White solid (177 mg, 66 %)

BSD 03 019 F2P31CPD CDC13 opt/topspin cristau 3



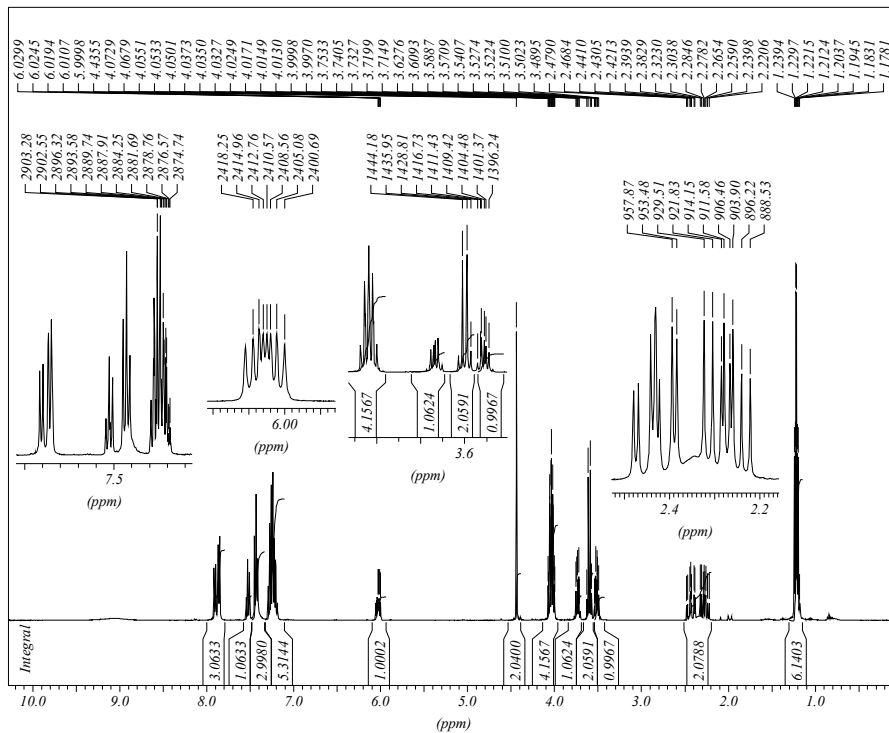
*** Current Data Parameters ***
 NAME : F2
 EXPNO : 203
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 23-48-43
 DATE_d : Mar 25 2009
 DE : 6.0 usec
 DS : 4
 NS : 16
 NUC1 : 31P
 O1 : 8098.78 Hz
 O2 : 1600.52 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
 RG : 4597.6000977
 SFO1 : 161.9836718 MHz
 SW : 200.4371 ppm
 SW_h : 32467.532 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 PC : 1.40
 SF : 161.975730 MHz
 *** ID NMR Plot Parameters ***
 Start : 150.22 ppm
 Stop : -50.22 ppm
 SR : 0.01 Hz
 ppm_cm : 9.50
 Hz_cm : 1538.75

(±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate **Bz-10c**



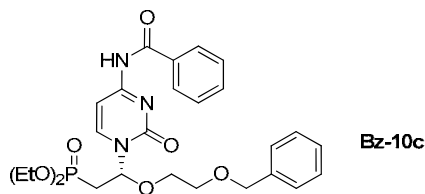
$C_{26}H_{32}N_3O_7P$
 MW = 529.53 g.mol⁻¹
 White solid (177 mg, 66 %)

BSD 03 019 F2PROTON CDCl3 opt/topspin cristau 3



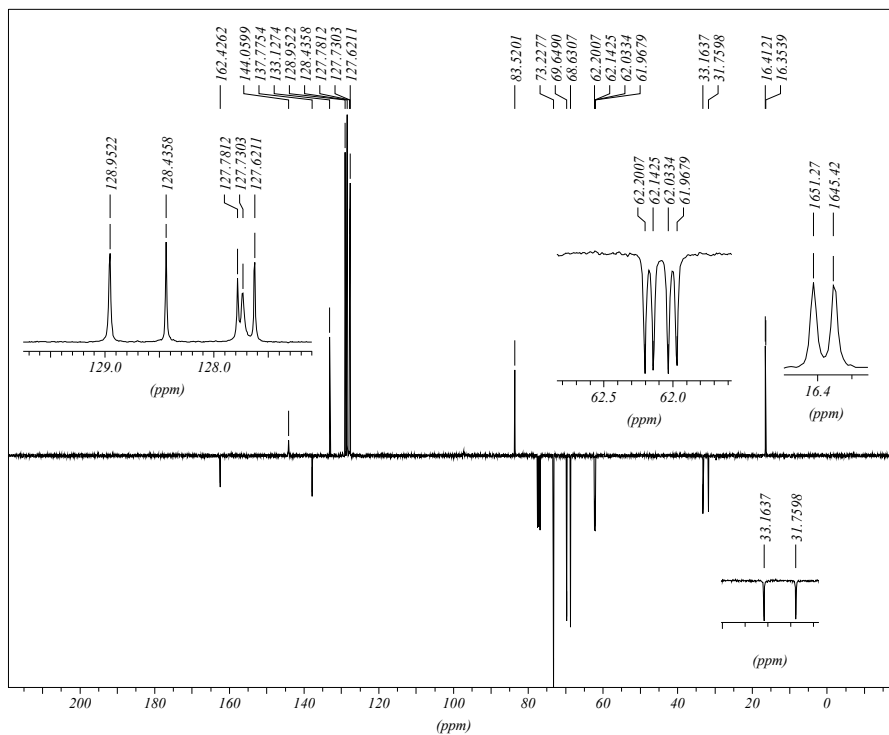
*** Current Data Parameters ***
 NAME : F2
 EXPNO : 204
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 23:51:52
 DATE_d : Mar 25 2009
 DE : 6.0 usec
 DS : 2
 NS : 16
 NUC1 : 1H
 O1 : 2400.78 Hz
 O2 : 2470.97 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
 RG : 57.000000
 SFO1 : 400.1324008 MHz
 SW : 14.9831 ppm
 SW_h : 5995.204 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 0.10 Hz
 PC : 1.00
 SF : 400.1300306 MHz
 *** ID NMR Plot Parameters ***
 Start : 10.28 ppm
 Stop : 0.04 ppm
 SR : 30.65 Hz
 ppm_cm : 0.49
 Hz_cm : 194.18

(±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate **Bz-10c**



$C_{26}H_{32}N_3O_7P$
MW = 529.53 g.mol⁻¹
White solid (177 mg, 66 %)

BSD 02 019 F2C13APT CDCl3 opt/topspin cristau 35



*** Current Data Parameters ***

NAME : F2
EXPNO : 210
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 15:27:19
DATE_d : Mar 26 2009
DE : 6.0 usec
DS : 4
NS : 1028
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 16384.000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

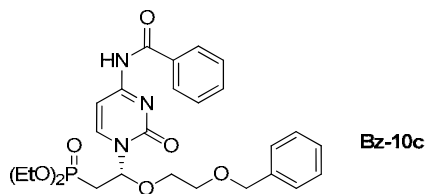
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

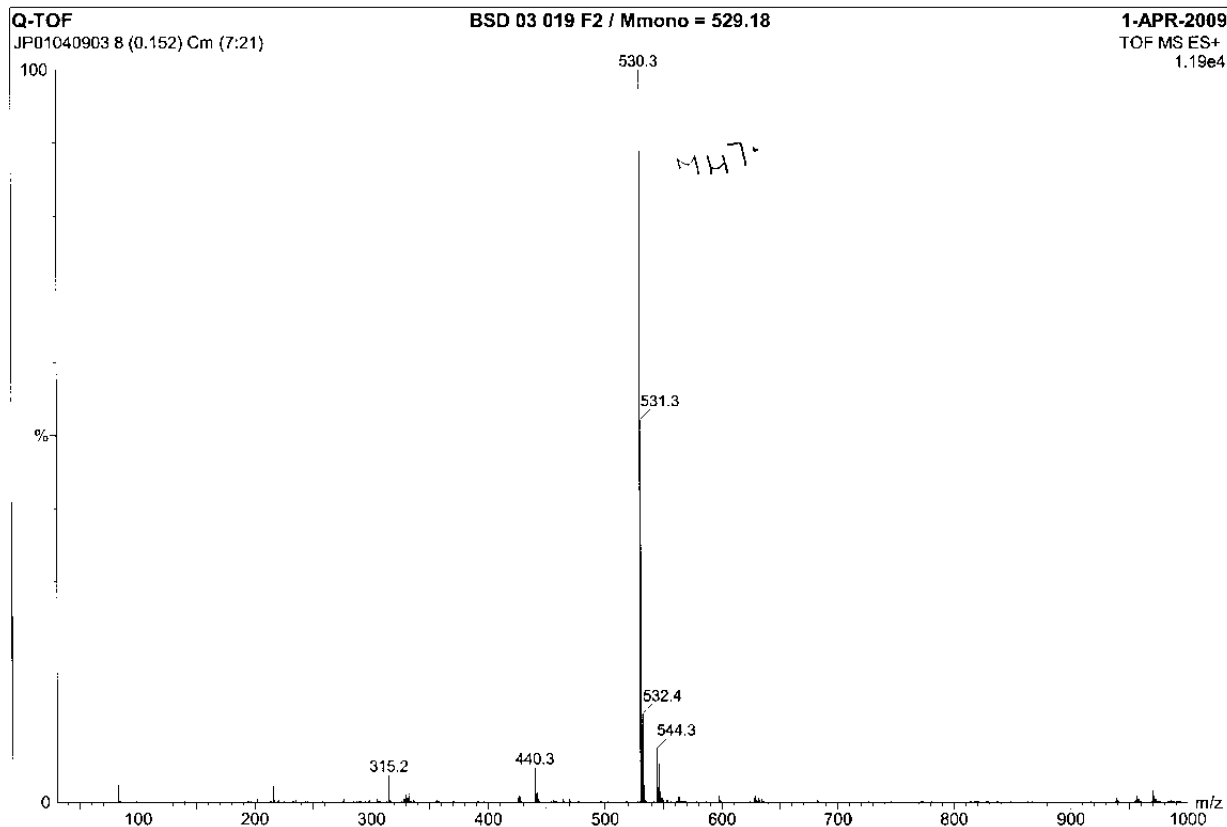
*** ID NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
ppm_cm : 11.30
Hz_cm : 1136.53

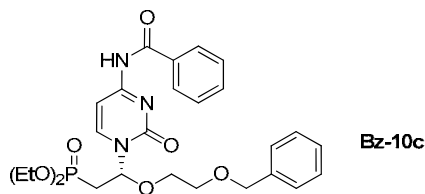
(±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate **Bz-10c**



$C_{26}H_{32}N_3O_7P$
MW = 529.53 g.mol⁻¹
White solid (177 mg, 66 %)



(±)-Diethyl 2-[2-(benzyloxy)ethoxy]-2-[2-(4-benzoylamino-2-oxo-2H-pyrimidin-1-yl)]ethylphosphonate **Bz-10c**



$C_{26}H_{32}N_3O_7P$
MW = 529.53 g.mol⁻¹
White solid (177 mg, 66 %)

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

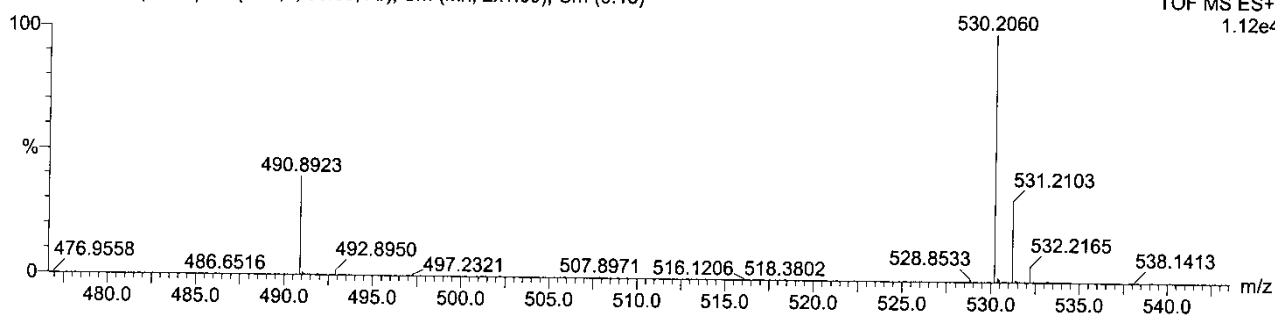
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

126 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

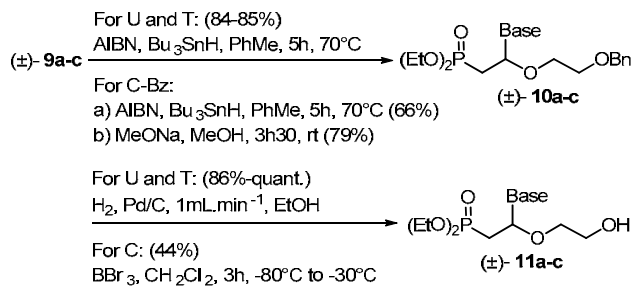
Q-TOF BSD 03 019 F2 / Mmono = 529.18
JP01040904 9 (0.170) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (9:15)

1-APR-2009
TOF MS ES+
1.12e4

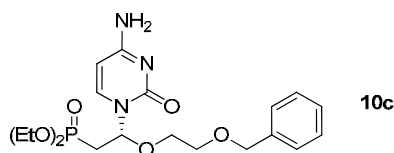


Minimum:				-5.0		
Maximum:		3.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
530.2060	530.2056	0.4	0.7	12.5	1	C26 H33 N3 O7 P

II.8. (±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate 10c

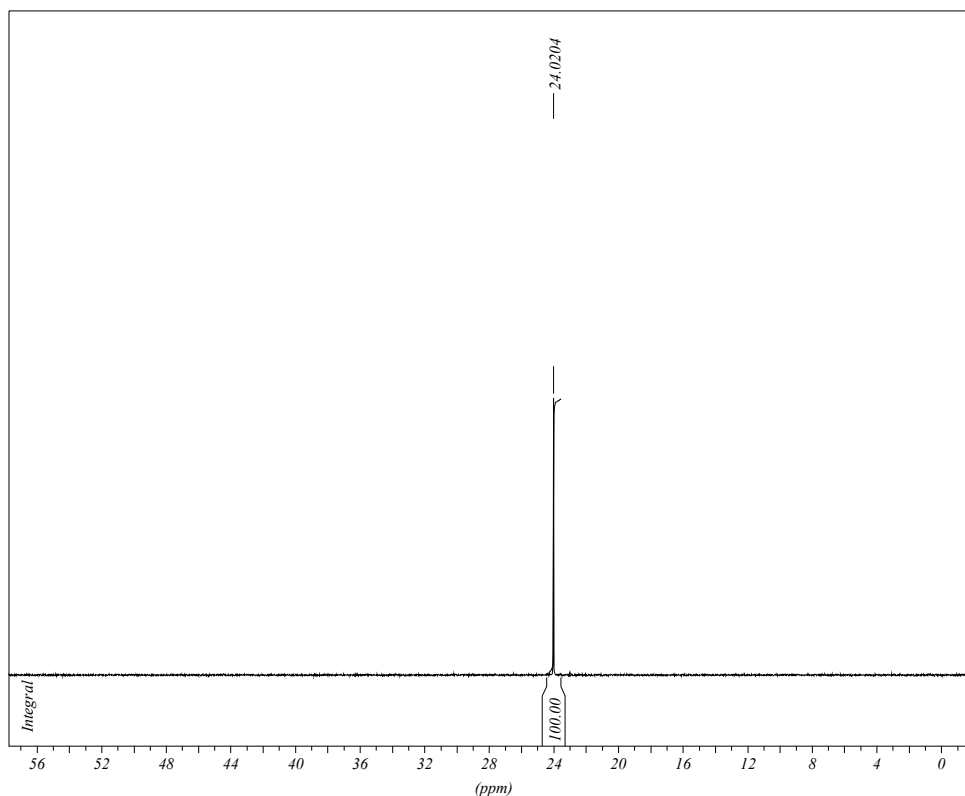


Scheme 3 Reduction and debenylation steps



C₁₉H₂₈N₃O₆P
 MW = 425.43 g.mol⁻¹
 Colorless oil (240 mg, 79 %)

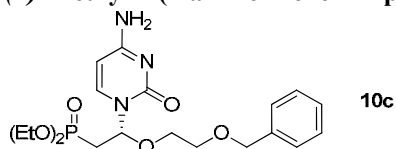
BSD 03 055 Orga 2P31CPD CDC13 opt/topspin cristau 55



*** Current Data Parameters ***

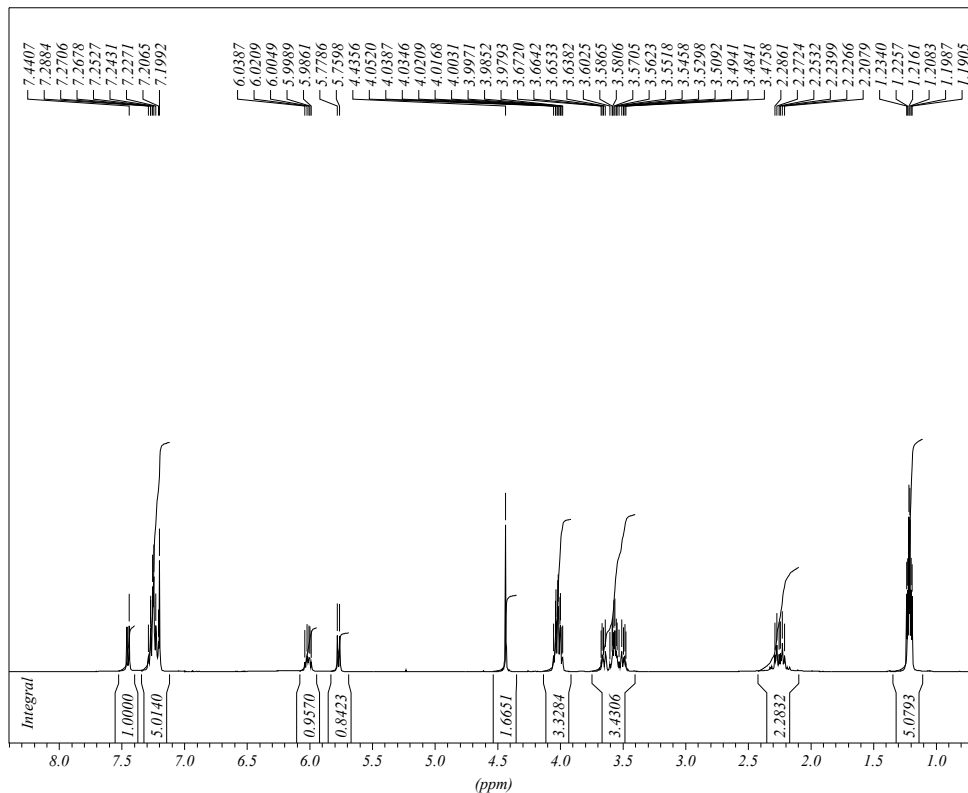
NAME : final
 EXPNO : 203
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_1 : 06:49:13
 DATE_d : May 13 2009
 NS : 16
 O1 : 8098.78 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
 RG : 20642.5000000
 SFO1 : 161.9836718 MHz
 SOLVENT : CDC13
 SW : 200.4371 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 57.74 ppm
 Stop : -2.29 ppm
 SR : 0.01 Hz
 SOLVENT : ?

(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate **10c**



$C_{19}H_{28}N_3O_6P$
MW = 425.43 g.mol⁻¹
Colorless oil (240 mg, 79 %)

BSD 03 055 Orga 2PROTON CDCl3 opt/topspin cristau 55



*** Current Data Parameters ***

NAME : final
EXPNO : 204
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 06:52:33
DATE_d : May 13 2009
NS : 16
O1 : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84
RG : 161.3000031
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 14.9831 ppm

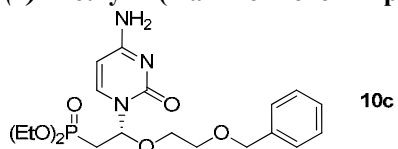
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

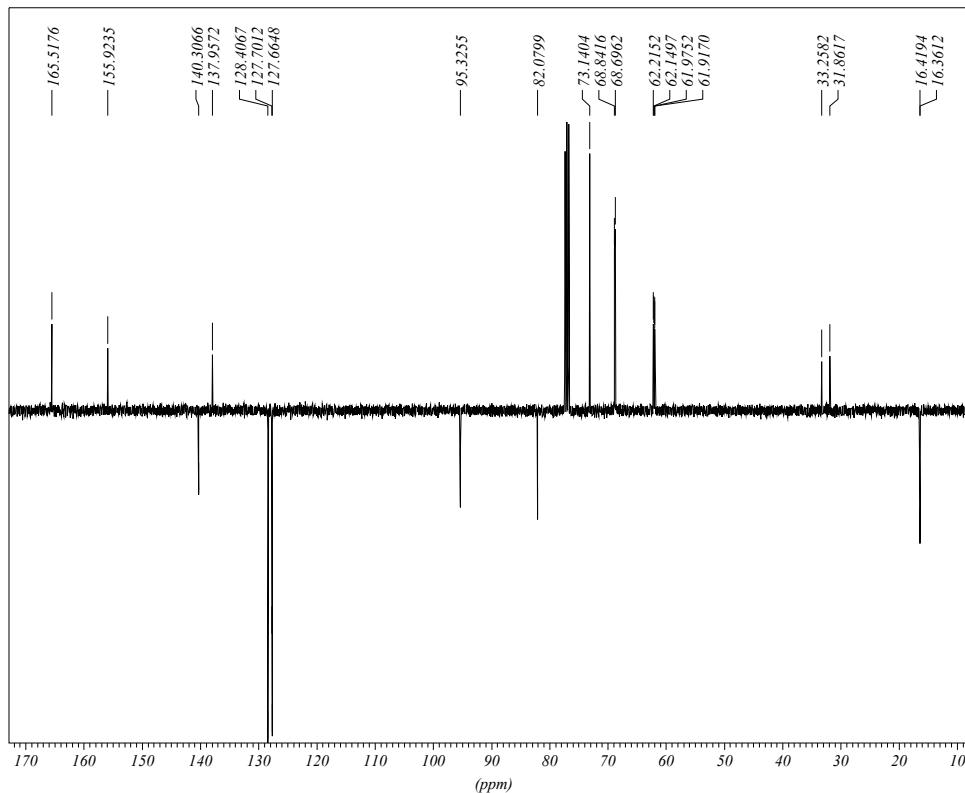
Start : 8.40 ppm
Stop : 0.66 ppm
SR : 34.45 Hz
SOLVENT : ?

(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate **10c**



$C_{19}H_{28}N_3O_6P$
MW = 425.43 g.mol⁻¹
Colorless oil (240 mg, 79 %)

BSD 03 055 Orga 2C13APT CDC13 opt/topspin cristau 55



*** Current Data Parameters ***

NAME : final
EXPNO : 205
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 13:00:17
DATE_d : May 13 2009
NS : 1024
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 16384.000000
SFO1 : 100.6228298 MHz
SOLVENT : CDC13
SW : 238.3238 ppm

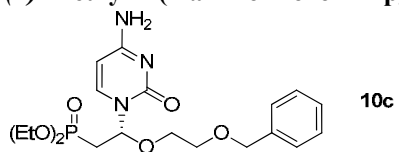
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

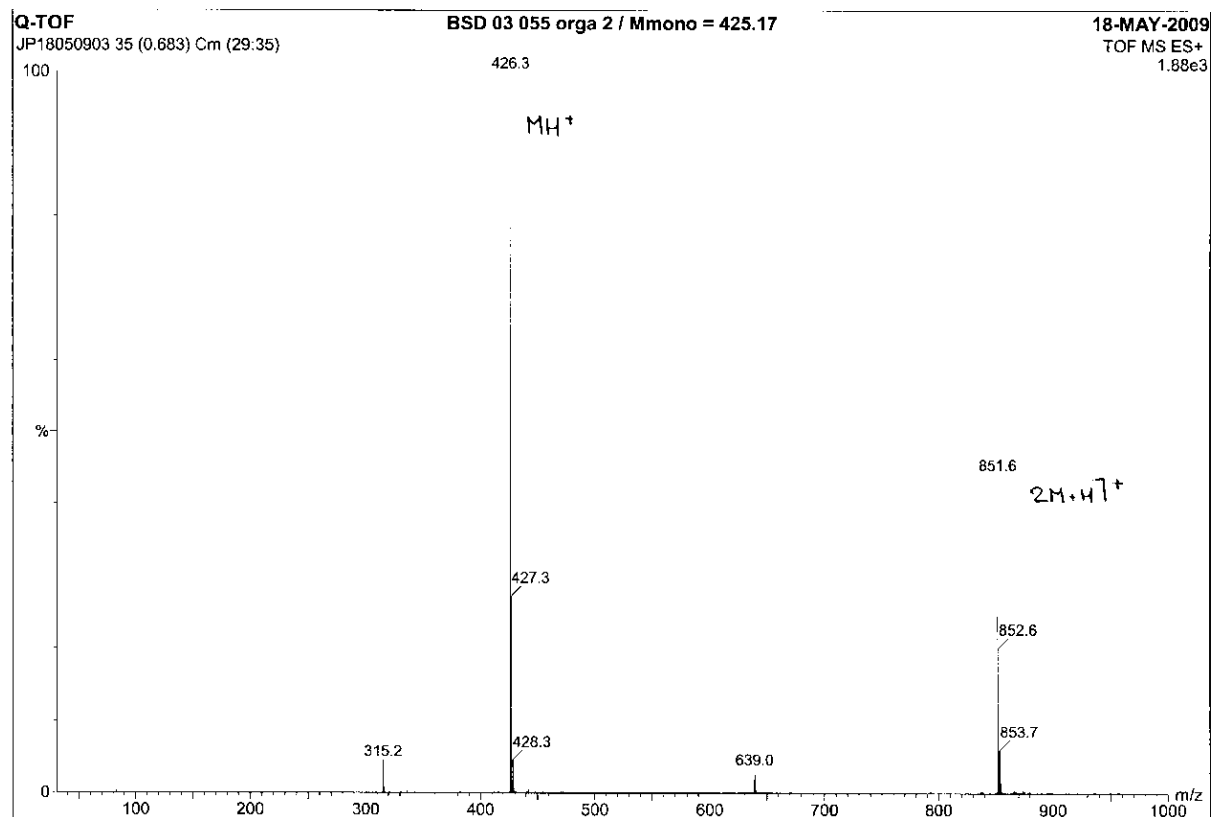
*** 1D NMR Plot Parameters ***

Start : 172.92 ppm
Stop : 6.37 ppm
SR : 0.00 Hz
SOLVENT : ?

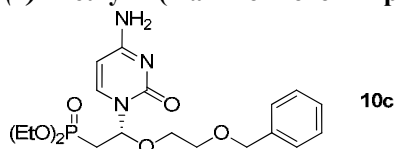
(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate **10c**



$C_{19}H_{28}N_3O_6P$
MW = 425.43 g.mol⁻¹
Colorless oil (240 mg, 79 %)



(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-[2-(benzyloxy)ethoxy]ethylphosphonate **10c**



$C_{19}H_{28}N_3O_6P$
MW = 425.43 g.mol⁻¹
Colorless oil (240 mg, 79 %)

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

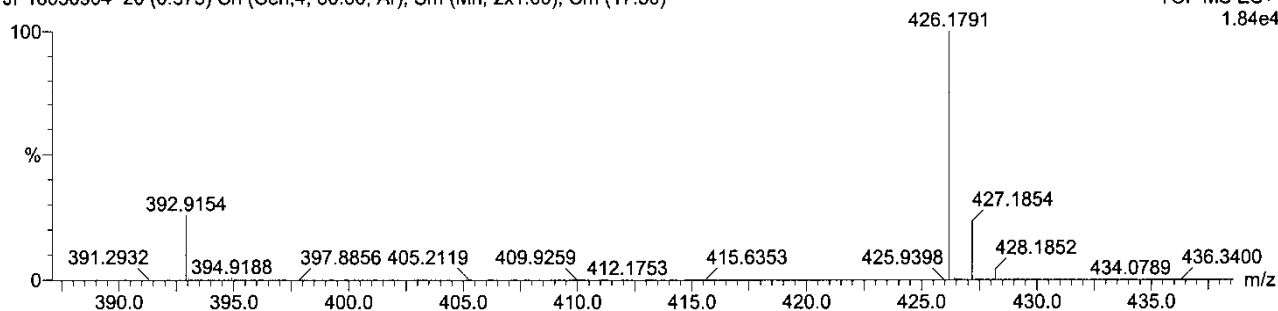
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

133 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 03 055 orga 2 / Mmono = 425.17
JP18050904 20 (0.375) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (17:50)

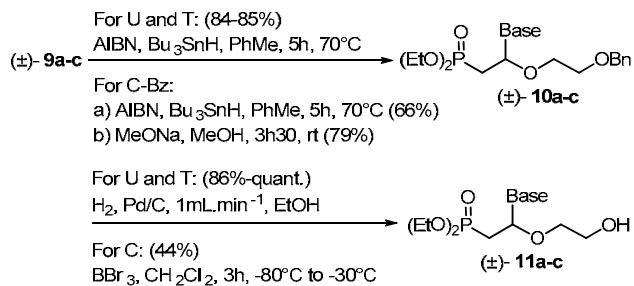
18-MAY-2009
TOF MS ES+
1.84e4



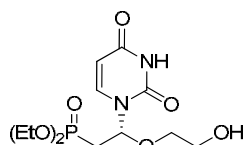
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
426.1791	426.1794	-0.3	-0.7	7.5	1	C19 H29 N3 O6 P ←
	426.1767	2.4	5.6	3.0	2	C16 H31 N2 O9 P

II.9. (±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



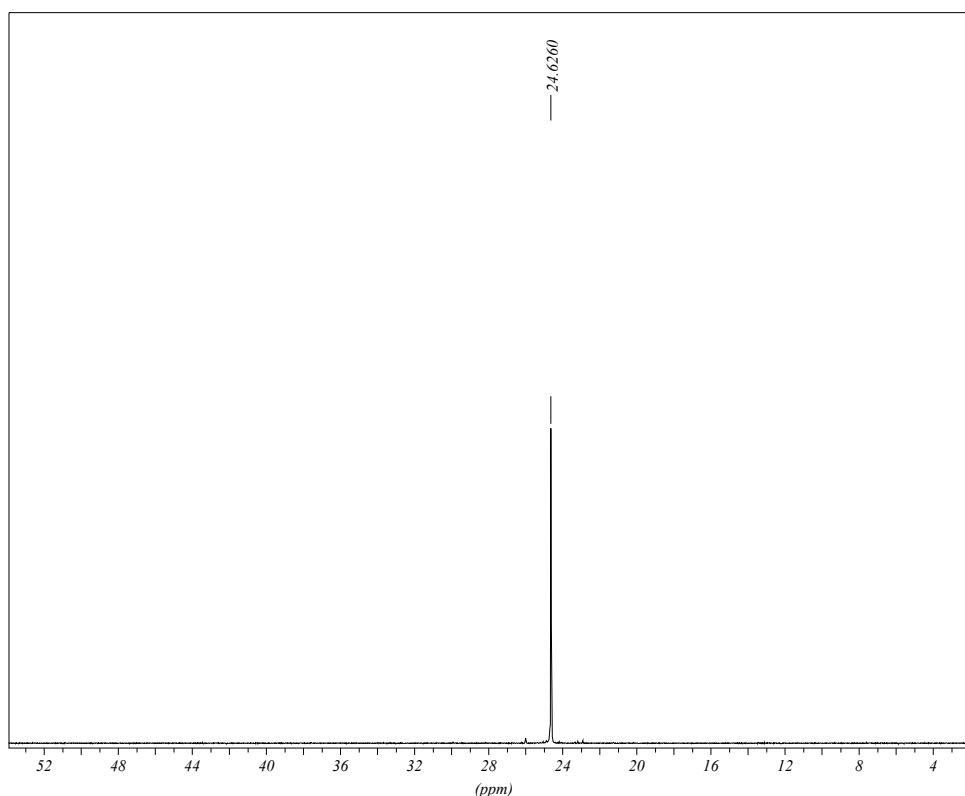
Scheme 3 Reduction and debenzylation steps



11a

C₁₂H₂₁N₂O₇P
MW = 336.28 g.mol⁻¹
Colorless (43 mg, quantitative yield)

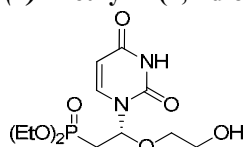
BSD 02 115-IP31CPD CDCl₃ opt/topspin cristau 34



*** Current Data Parameters ***

NAME : FULLH2-1
EXPNO : 201
PROCNO : 0
*** Acquisition Parameters ***
DATE_1 : 07:33:45
DATE_d : Feb 02 2009
NS : 16
O1 : 8098.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 10321.2998047
SFO1 : 161.9836718 MHz
SOLVENT : CDCl₃
SW : 200.4371 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 53.90 ppm
Stop : 1.56 ppm
SR : 0.01 Hz
SOLVENT : ?

(±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



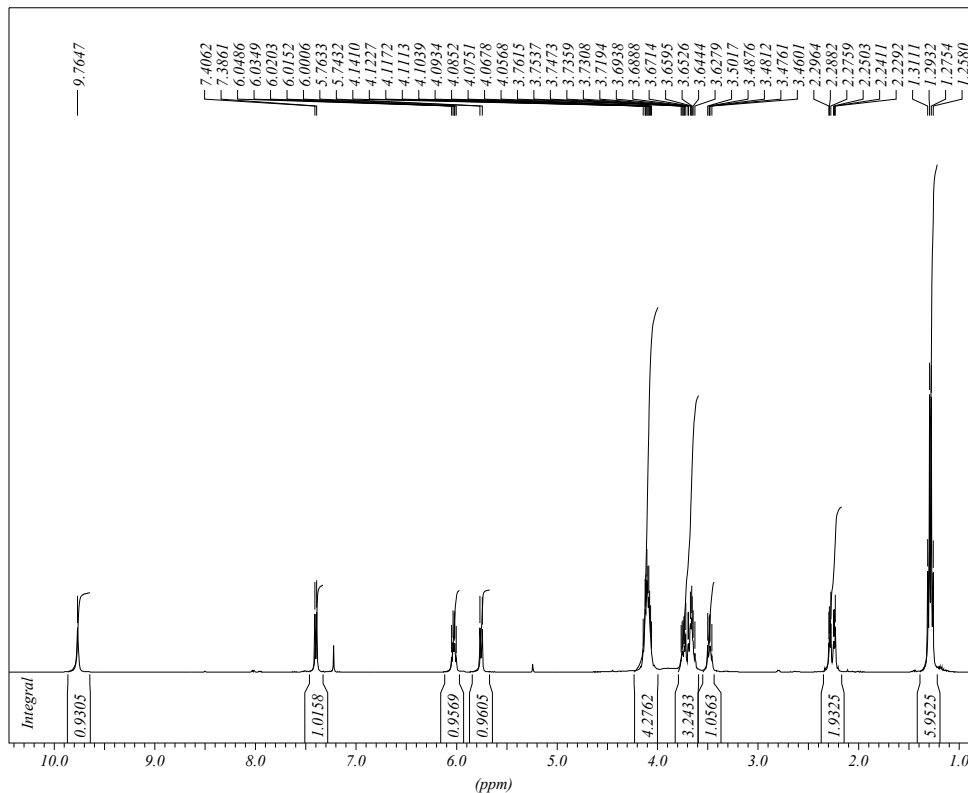
11a

C₁₂H₂₁N₂O₇P

MW = 336.28 g.mol⁻¹

Colorless (43 mg, quantitative yield)

BSD 02 115-2PROTON CDCl3 opt/topspin cristau 28



*** Current Data Parameters ***

NAME : sech2
EXPNO : 201
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 03:37:52
DATE_d : Feb 13 2009
NS : 16
O1 : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 80.5999985
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 14.9831 ppm

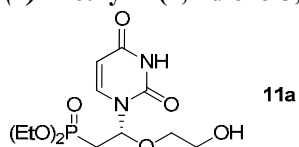
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

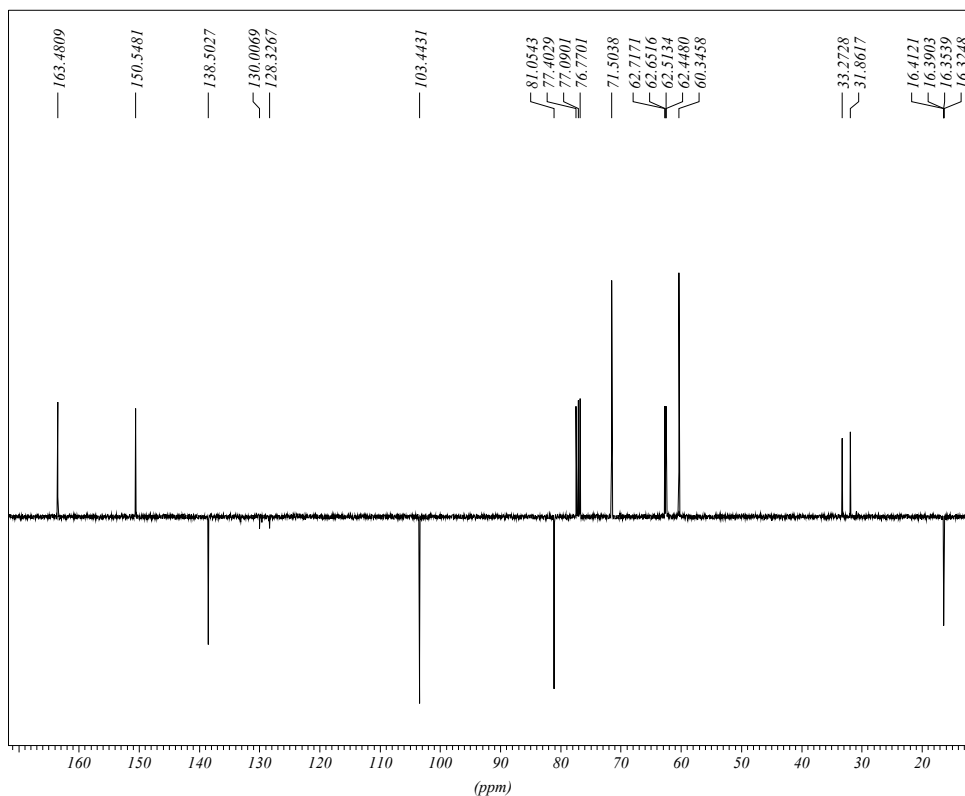
Start : 10.45 ppm
Stop : 0.81 ppm
SR : 26.28 Hz
SOLVENT : ?

(±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



$C_{12}H_{21}N_2O_7P$
MW = 336.28 g.mol⁻¹
Colorless (43 mg, quantitative yield)

BSD 02 115C13APT CDC13 opt/topspin cristau 14



*** Current Data Parameters ***

NAME : 13C
EXPNO : 230
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 13:57:25
DATE_d : Feb 19 2009
NS : 1024
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 16384.000000
SFO1 : 100.6228298 MHz
SOLVENT : CDC13
SW : 238.3238 ppm

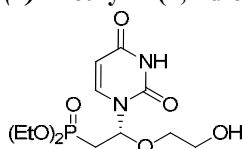
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 171.71 ppm
Stop : 10.80 ppm
SR : 0.00 Hz
SOLVENT : ?

(±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



11a

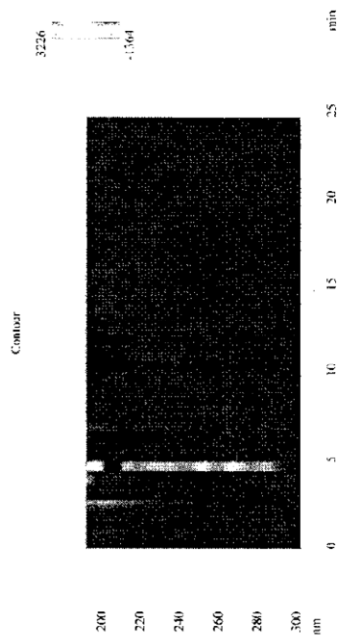
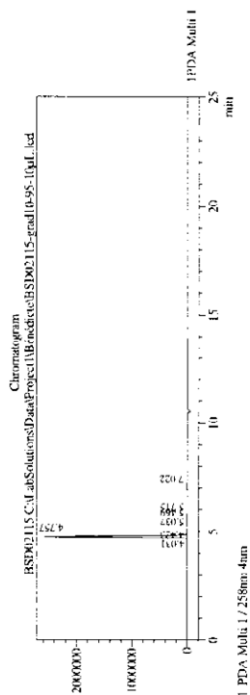
C₁₂H₂₁N₂O₇P

MW = 336.28 g.mol⁻¹

Colorless (43 mg, quantitative yield)

23/02/2009 11:33:16 2 / 2

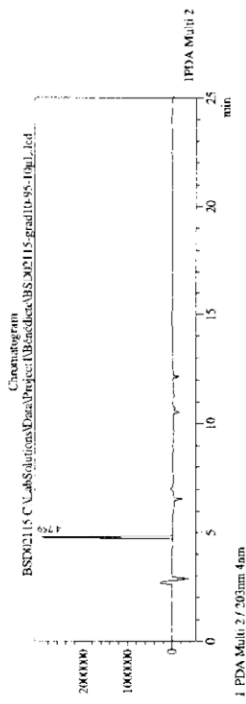
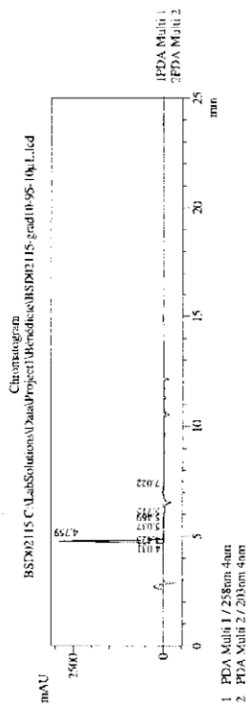
PDA Chl 25Scan-4nm		PeakTable	
Pic	Temp (st.)	Hauteur	Area %
1	4.031	1978	0.075
2	4.423	8898	0.339
3	4.757	2576466	97.028
4	5.037	5318	0.202
5	5.409	6471	0.246
6	5.715	8615	0.328
7	7.022	18459	0.700
Total		2623510	100.000



23/02/2009 11:33:16 1 / 2

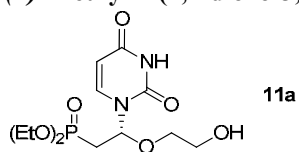
==== Shimadzu LCsolution Analysis Report =====

C:_Data\Project1\Benedicte\BSD02115.grad\0-95-10µL.cd
 Acquired by Admin
 Sample Name BSD02115
 Sample ID BSD02115
 Tray# 91
 Injection Volume 10 µL
 Data File Name BSD02115.grad\0-95-10µL.cd
 Method File Name Methode_Pierce_21cm
 Batch File Name 23.02.09 (BSD02115-117).lib
 Report File Name bene_report1.r
 Data Acquired 23/02/2009 10:26:40
 Data Processed 23/02/2009 11:29:35

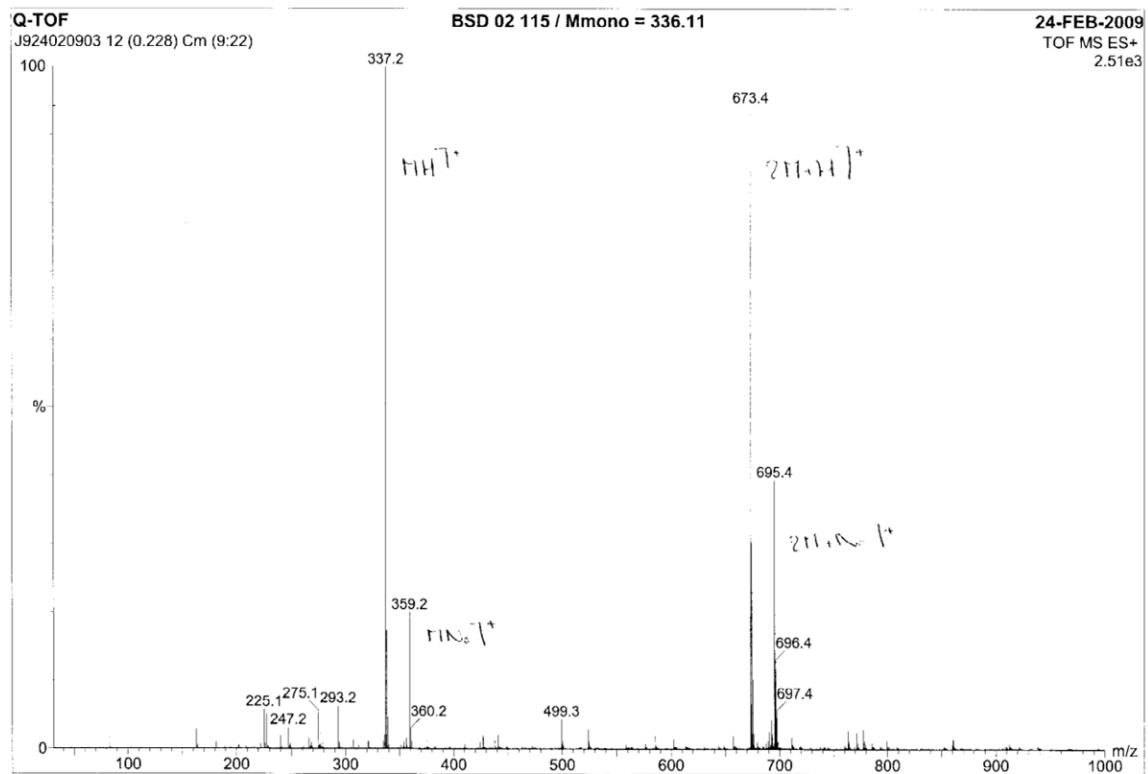


PDA Chl 20Scan-4nm		PeakTable	
Pic	Temp (st.)	Hauteur	Area %
1	4.757	2948934	100.000
Total		2948934	100.000

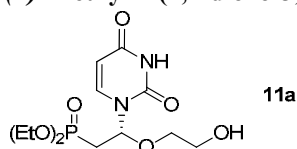
(±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



$C_{12}H_{21}N_2O_7P$
MW = 336.28 g.mol⁻¹
Colorless (43 mg, quantitative yield)



(±)-Diethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11a**



$C_{12}H_{21}N_2O_7P$
MW = 336.28 g.mol⁻¹
Colorless (43 mg, quantitative yield)

Elemental Composition Report

Page 1

Single Mass Analysis

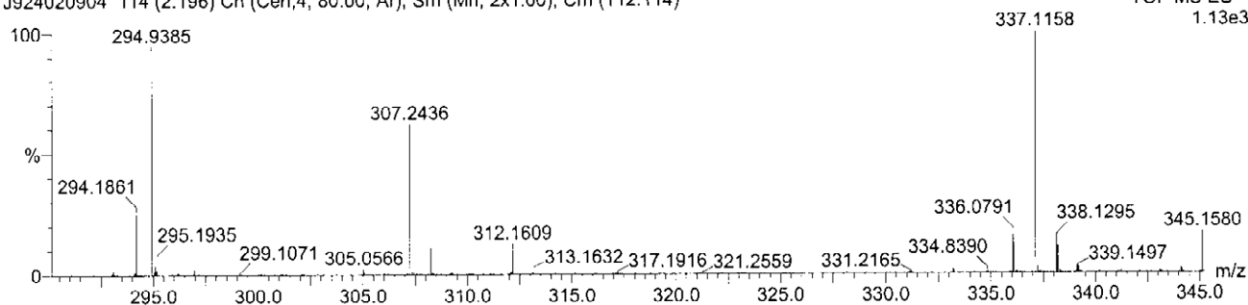
Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

98 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 02 115 / Mmono = 336.11
J924020904 114 (2.196) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (112:114)

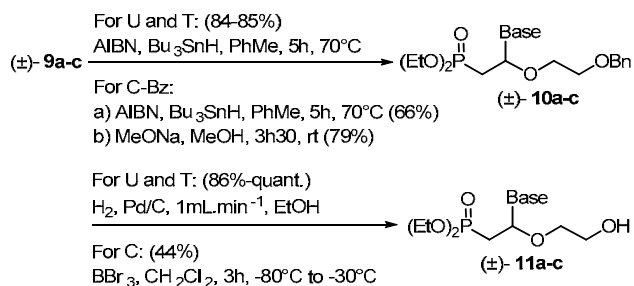
24-FEB-2009
TOF MS ES+
1.13e3



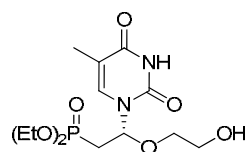
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
337.1158	337.1165	-0.7	-2.0	3.5	1	C12 H22 N2 O7 P
	337.1151	0.7	2.0	4.0	2	C10 H20 N5 O6 P
	337.1138	2.0	6.0	-1.0	3	C9 H24 N O10 P

II.10. (±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



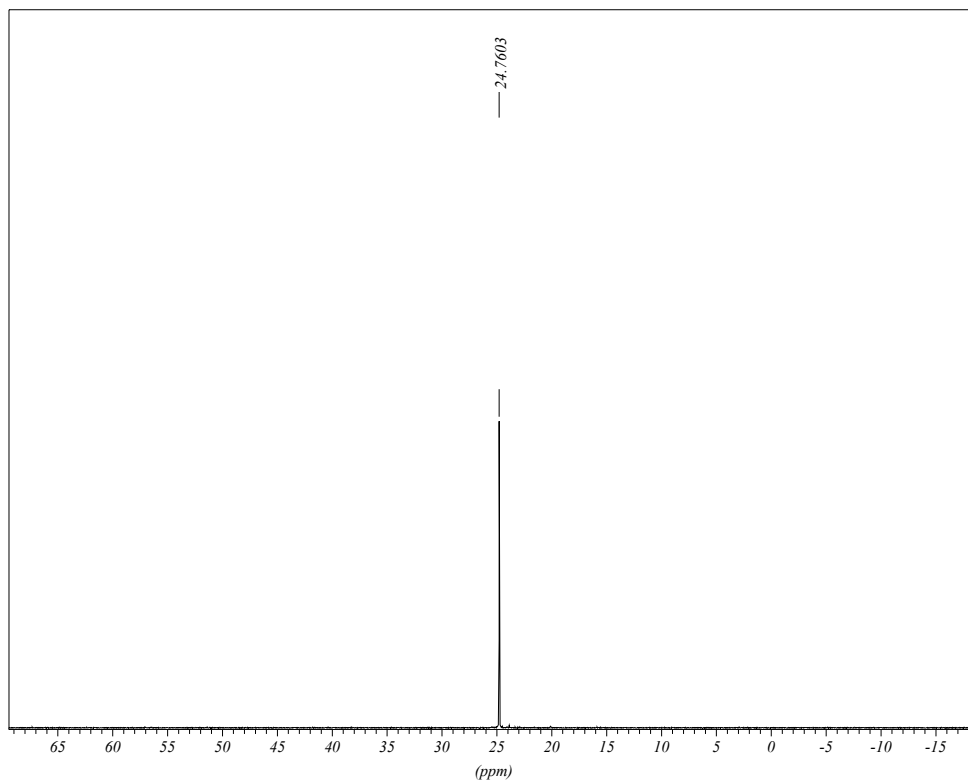
Scheme 3 Reduction and debenzylation steps



11b

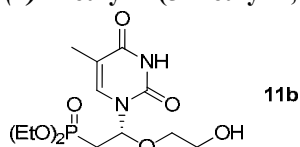
C₁₃H₂₃N₂O₇P
 MW = 350.31 g.mol⁻¹
 Colorless oil (33 mg, 86 %)

bsd02117-2eme hydrogenolyse
 31P CPD CDC13



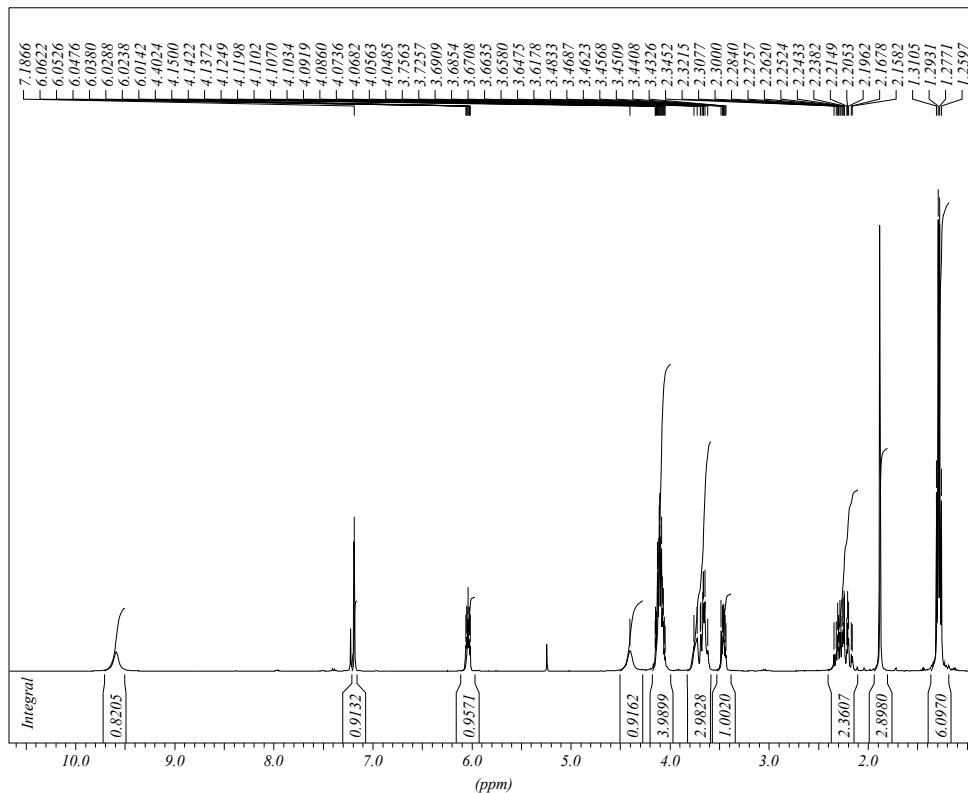
*** Current Data Parameters ***
 NAME : exp2
 EXPNO : 221
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_1 : 09-17-50
 DATE_d : Feb 05 2009
 NS : 16
 O1 : -5062.76 Hz
 PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
 RG : 10321.2998047
 SFO1 : 101.2494172 MHz
 SOLVENT : CDC13
 SW : 401.4878 ppm
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 69.44 ppm
 Stop : -18.87 ppm
 SR : 0.00 Hz
 SOLVENT : ?

(±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



$C_{13}H_{23}N_2O_7P$
MW = 350.31 g.mol⁻¹
Colorless oil (33 mg, 86 %)

BSD 02 117-2PROTON CDCl3 opt/topspin cristau 29



*** Current Data Parameters ***

NAME : sech1

EXPNO : 202

PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03:45:15

DATE_d : Feb 13 2009

NS : 16

O1 : 2400.78 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G

RG : 71.8000031

SFO1 : 400.1324008 MHz

SOLVENT : CDCl3

SW : 14.9831 ppm

*** Processing Parameters ***

GB : 0.0000000

LB : 1.00 Hz

SI : 32768

TI :

*** 1D NMR Plot Parameters ***

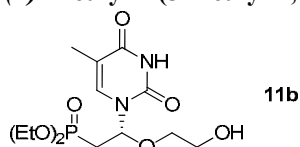
Start : 10.67 ppm

Stop : 0.89 ppm

SR : 25.24 Hz

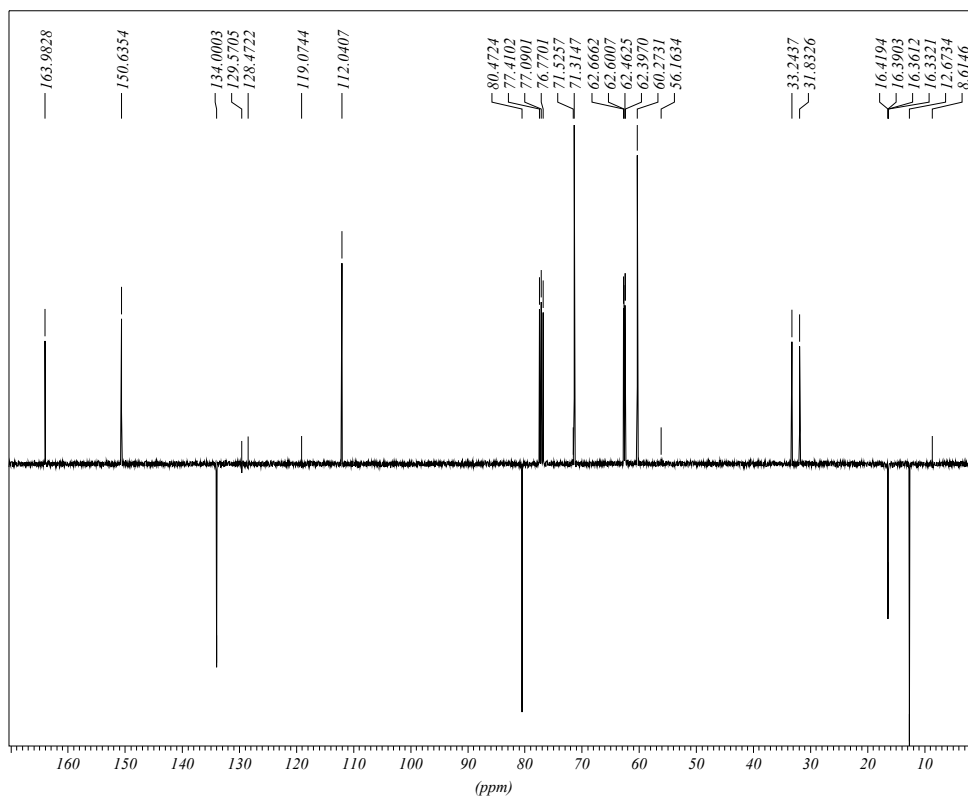
SOLVENT : ?

(±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



$C_{13}H_{23}N_2O_7P$
MW = 350.31 g.mol⁻¹
Colorless oil (33 mg, 86 %)

BSD 02 117C13APT CDC13 opt/topspin cristau 15



*** Current Data Parameters ***

NAME : 13C
EXPNO : 231
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 15:01:56
DATE_d : Feb 19 2009
NS : 1024
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 16384.000000
SFO1 : 100.6228298 MHz
SOLVENT : CDC13
SW : 238.3238 ppm

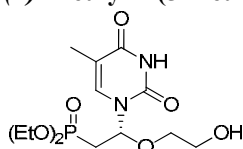
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 170.37 ppm
Stop : 0.58 ppm
SR : 0.00 Hz
SOLVENT : ?

(±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



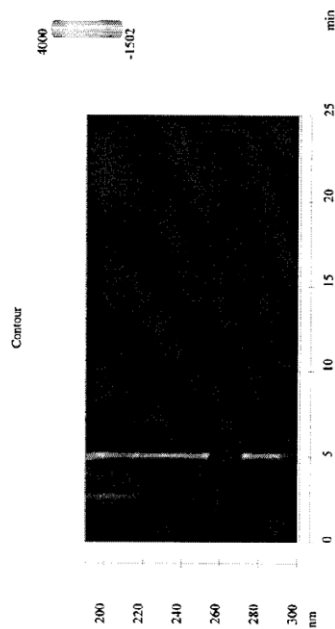
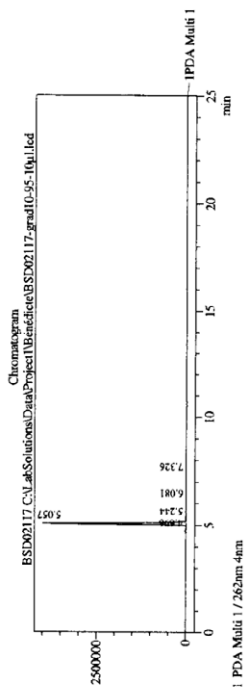
11b

C₁₃H₂₃N₂O₇P
 MW = 350.31 g.mol⁻¹
 Colorless oil (33 mg, 86 %)

23/02/2009 11:34:50 2 / 2

PeakTable

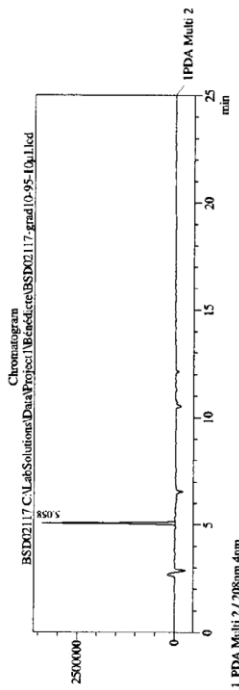
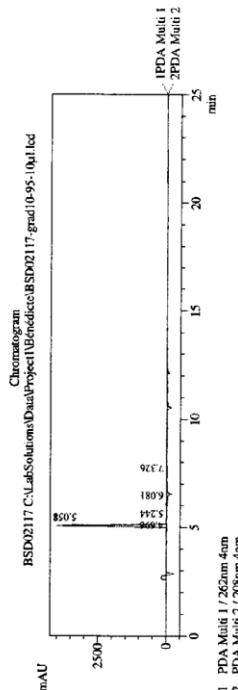
Pic	Temps ret.	Hauteur	Aire	% Hauteur	Area %
1	4.696	19675	165944	0.487	1.020
2	5.057	3997370	16381558	98.584	98.279
3	5.244	6741	48205	0.167	0.289
4	6.081	567	26447	0.166	0.171
5	7.326	3317	6840	0.196	0.242
Total		4038389	1666484	100.000	100.000



23/02/2009 11:34:50 1 / 2

==== Shimadzu LCsolution Analysis Report =====

C:\LabSolutions\Data\Project1\Bénédictie\BSD02117_grad10-95-10µl.lcd
 Acquired by :
 Sample Name : BSD02117
 Sample ID : BSD02117
 Tray# : 1
 Vial # : 92
 Injection Volume : 10 µl
 Data File Name : BSD02117_grad10-95-10µl.lcd
 Method File Name : Methode Pierre 2.lcm
 Batch File Name : 23-02-09 (BSD02115-117).lcb
 Report File Name : bene_report.rpt
 Data Acquired : 23/02/2009 10:52:07
 Data Processed : 23/02/2009 11:34:06



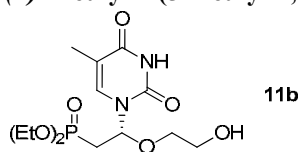
PeakTable

Pic	Temps ret.	Hauteur	Aire	% Hauteur	Area %
1	5.058	3418065	15656462	100.000	100.000
Total		3418065	15656462	100.000	100.000

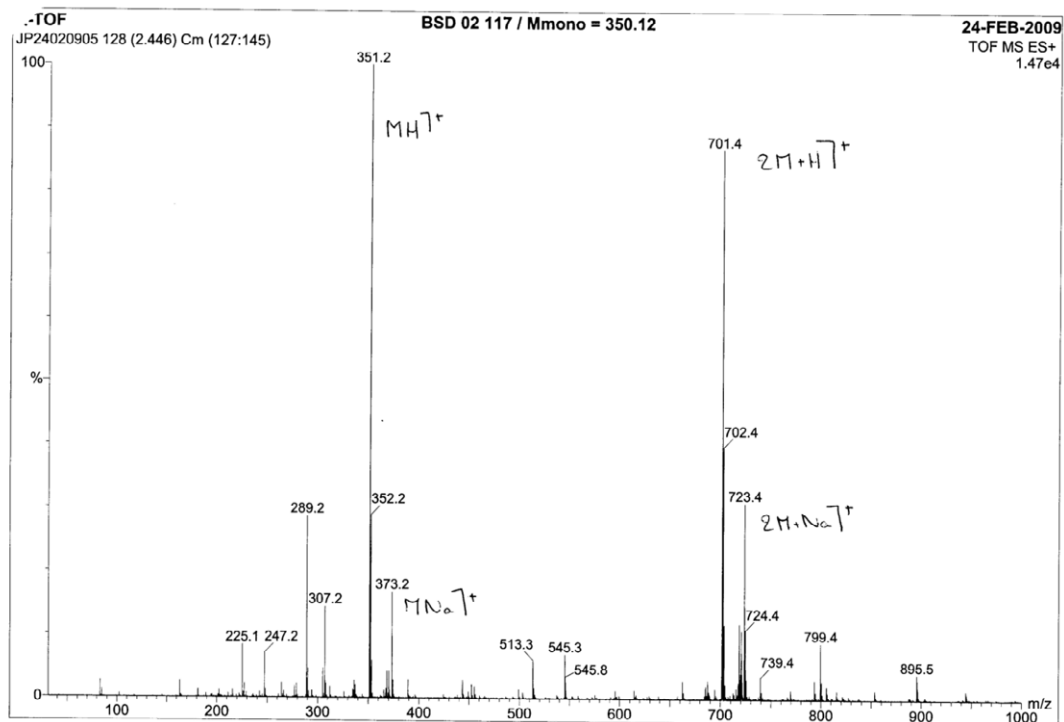
C:\LabSolutions\Data\Project1\Bénédictie\BSD02117_grad10-95-10µl.lcd

C:\LabSolutions\Data\Project1\Bénédictie\BSD02117_grad10-95-10µl.lcd

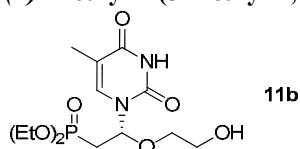
(±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



$C_{13}H_{23}N_2O_7P$
MW = 350.31 g.mol⁻¹
Colorless oil (33 mg, 86 %)



(±)-Diethyl 2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11b**



$C_{13}H_{23}N_2O_7P$
MW = 350.31 g.mol⁻¹
Colorless oil (33 mg, 86 %)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

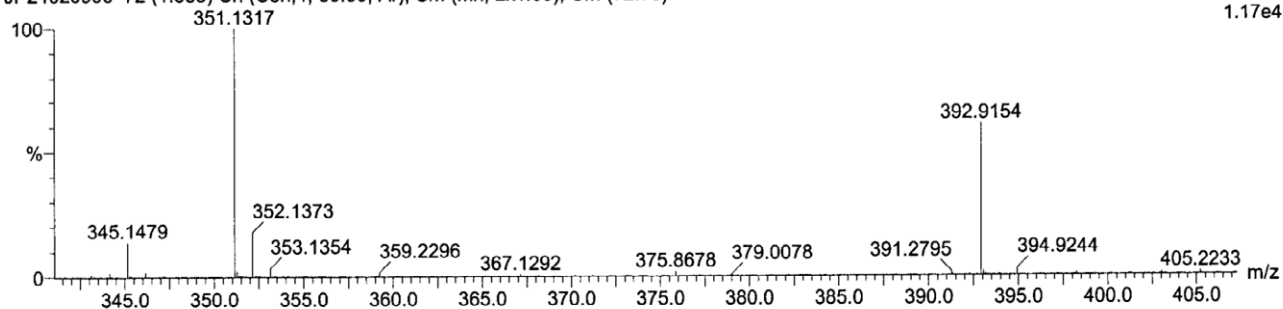
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

104 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF BSD 02 117 / Mmono = 350.12
JP24020906 72 (1.383) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (72:78)

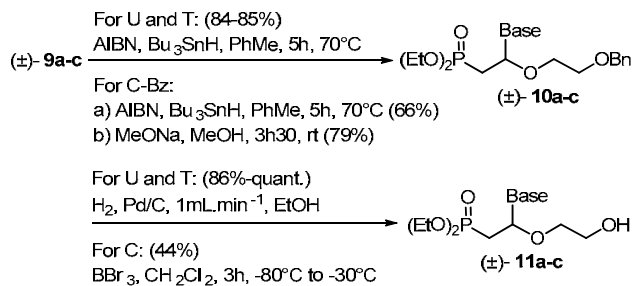
24-FEB-2009
TOF MS ES+
1.17e4



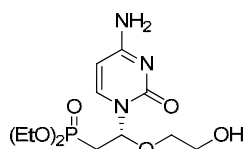
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
351.1317	351.1321	-0.4	-1.2	3.5	1	C13 H24 N2 O7 P
	351.1308	0.9	2.6	4.0	2	C11 H22 N5 O6 P
	351.1294	2.3	6.5	-1.0	3	C10 H26 N O10 P

II.11. (±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**

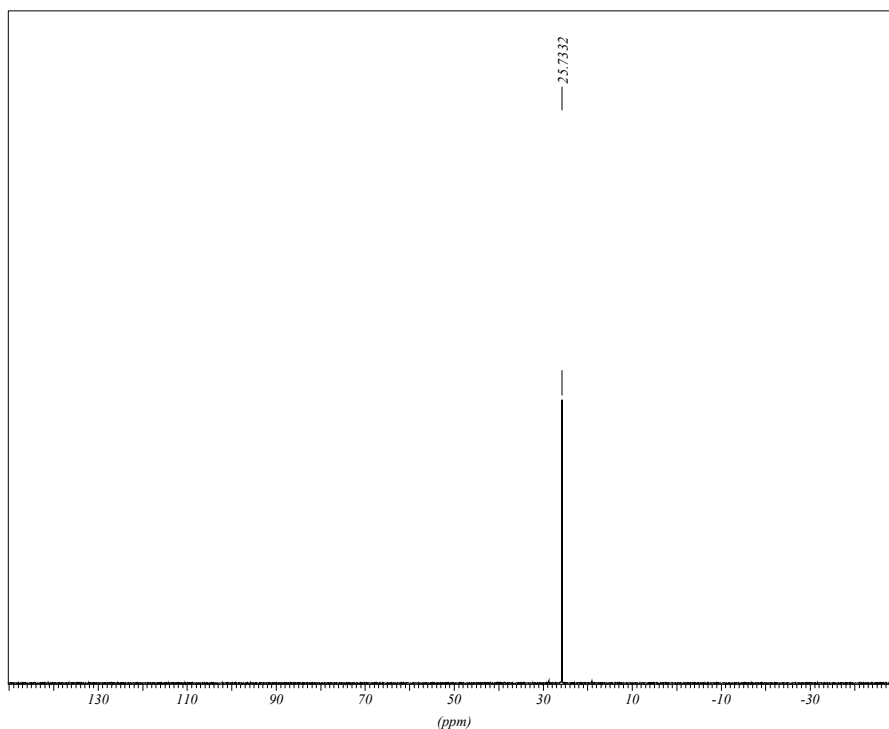


Scheme 3 Reduction and debenzylation steps



C₁₂H₂₂N₃O₆P
 MW = 335.36 g.mol⁻¹
 Colorless oil (35 mg, 37 %)

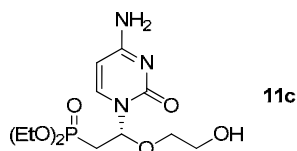
BSD 03 093 CIP3ICPD CDC13 opt/topspin cristau 46



```

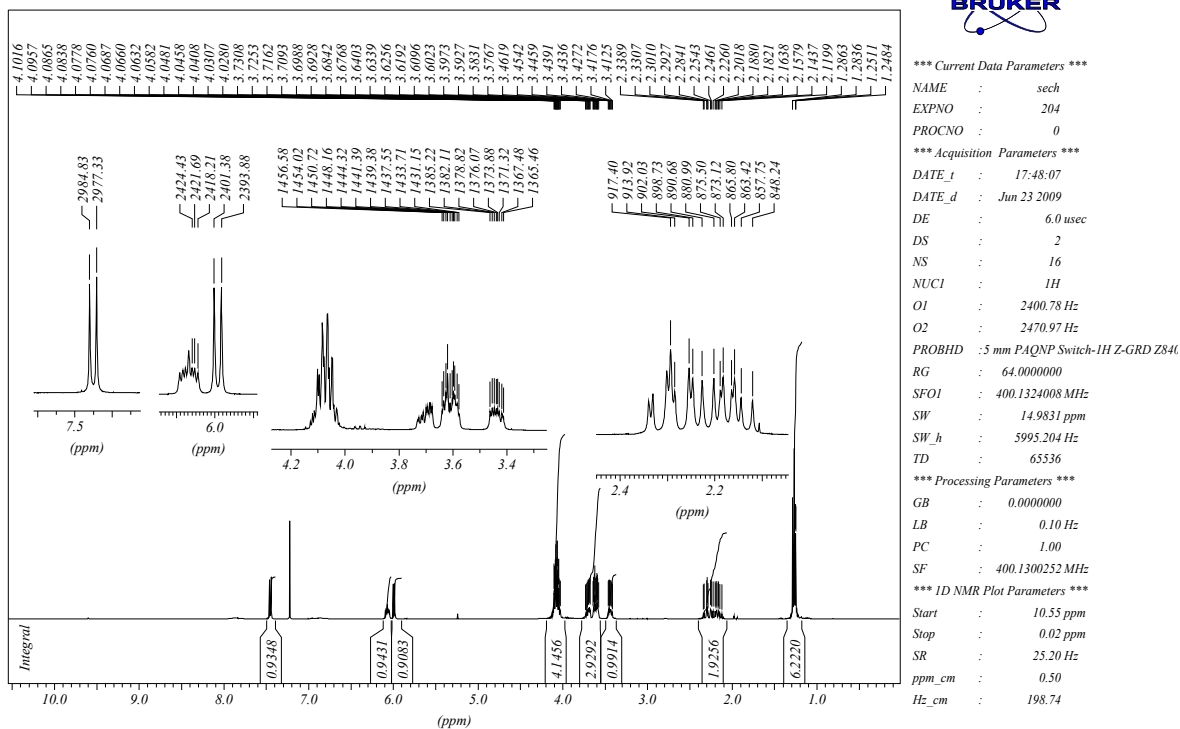
*** Current Data Parameters ***
NAME      : 203
EXPNO     : 31
PROCNO    : FID
*** Acquisition Parameters ***
DATE_t    : 17:44:56
DATE_d    : Jun 23 2009
DE        : 6.0 usec
DS        : 4
NS        : 16
NUC1      : 31P
O1        : 8098.78 Hz
O2        : 1600.52 Hz
PROBHD    : 5 mm PAQNP Switch-1H Z-GRD Z84L
RG        : 11585.2001953
SFO1      : 161.9836718 MHz
SW        : 200.4371 ppm
SW_h      : 32467.532 Hz
TD        : 65536
*** Processing Parameters ***
GB        : 0.0000000
LB        : 1.00 Hz
PC        : 1.40
SF        : 161.9755730 MHz
*** 1D NMR Plot Parameters ***
Start     : 150.22 ppm
Stop      : -50.22 ppm
SR        : 0.01 Hz
ppm_cm    : 9.46
Hz_cm     : 1531.49
    
```

(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**

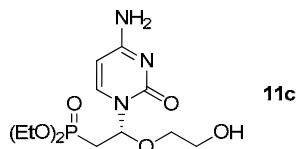


$C_{12}H_{22}N_3O_6P$
 MW = 335.36 g.mol⁻¹
 Colorless oil (35 mg, 37 %)

BSD 03 093 CIPROTON CDC13 opt/topspin cristau 46

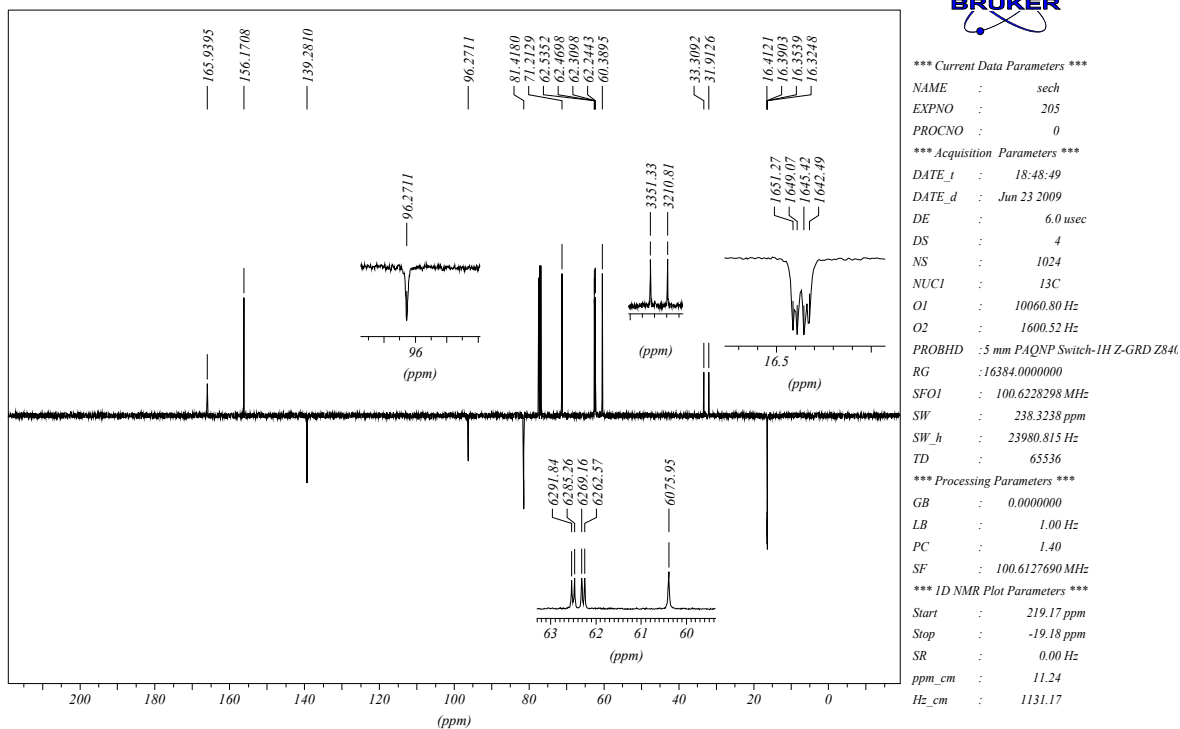


(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**

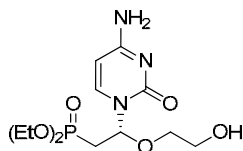


$C_{12}H_{22}N_3O_6P$
MW = 335.36 g.mol⁻¹
Colorless oil (35 mg, 37 %)

BSD 03 093 C1C13APT CDCl3 opt/topspin cristau 46



(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**



11c

$C_{12}H_{22}N_3O_6P$
 MW = 335.36 g.mol⁻¹
 Colorless oil (35 mg, 37 %)

01/07/2009 15:23:42 3

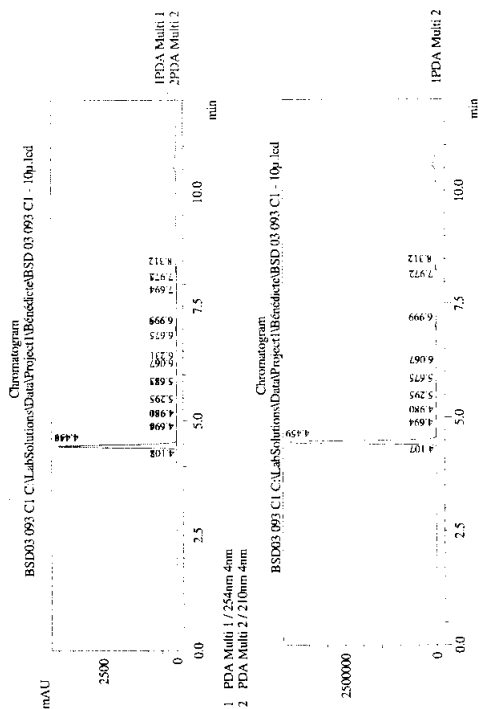
PeakTable

Pic	Temps ret.	Hauteur	Area	% Hauteur	Area %
1	4.108	2517	13375	0.062	0.092
2	4.448	3995832	14202862	96.498	97.935
3	4.696	10605	46684	0.281	0.188
4	4.980	4983	36705	0.120	0.252
5	5.295	10093	80747	0.246	0.553
6	5.683	2030	9104	0.050	0.062
7	6.231	1678	8058	0.041	0.055
8	6.675	17900	127447	0.441	0.874
9	6.995	1773	11811	0.044	0.081
10	7.694	3424	18908	0.084	0.130
11	7.975	4056712	14589380	100.000	100.000
Total					

01/07/2009 15:23:42 1

==== Shimadzu LCsolution Analysis Report =====

C:\...Project1\Bénédicté\BSD 03 093 C1 - 10µ.lcd
 Acquired by : Admin
 Sample Name : BSD03 093 C1
 Sample ID : BSD03 093 C1
 Tray # : 1
 Vial # : 98
 Injection Volume : 10 µl
 Data File Name : BSD 03 093 C1 - 10µ.lcd
 Method Name : Multi Peaks 2 exs 7
 Report File Name : 26_06_09_09rad\0-98-2.lcd
 Report File Path : Default.lcd
 Data Acquired : 26/06/2009 14:52:38
 Data Processed : 01/07/2009 15:22:49



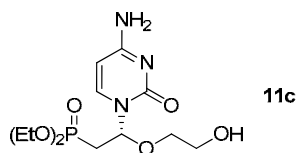
PeakTable

Pic	Temps ret.	Hauteur	Area	% Hauteur	Area %
1	4.107	4181	19865	0.101	0.101
2	4.459	3995837	18667404	96.579	95.347
3	4.694	24540	135037	0.593	0.690
4	4.980	13105	62490	0.317	0.319
5	5.295	10763	74375	0.360	0.380
6	5.675	14953	32444	0.171	0.177
7	6.099	26395	185910	0.156	1.001
8	6.672	6126	73384	0.148	0.375
9	6.972	34320	196301	0.834	1.063
10	8.312	4137390	19578423	100.000	100.000
Total					

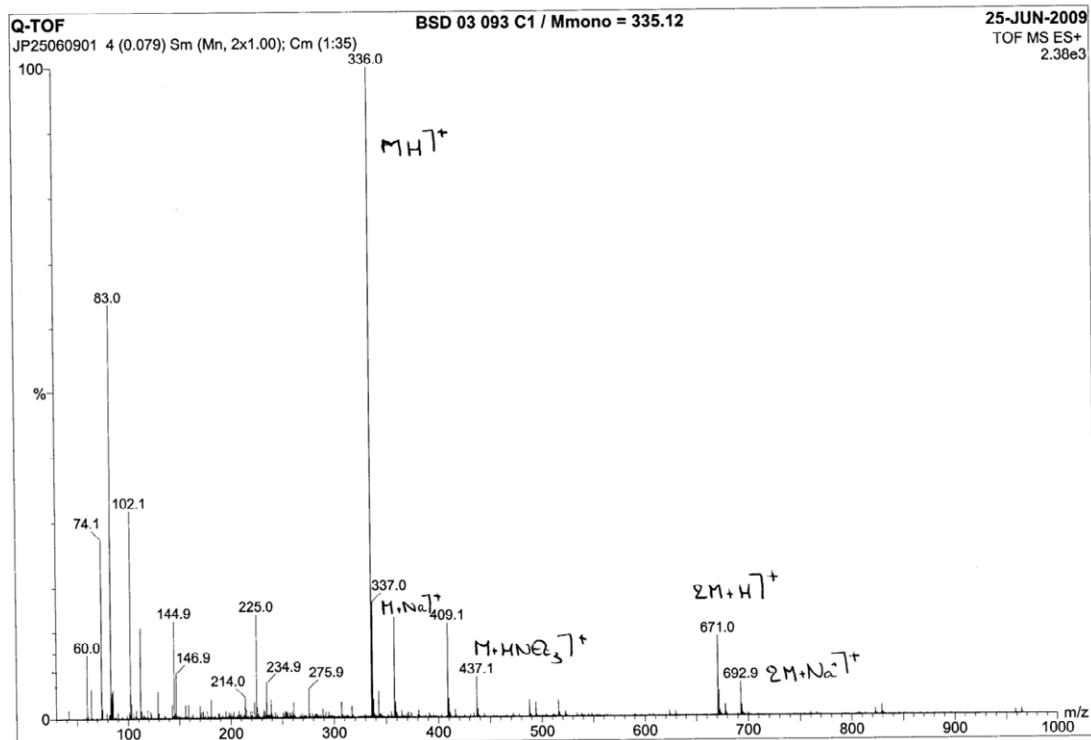
C:\LabSolutions\Data\Project1\Bénédicté\BSD 03 093 C1 - 10µ.l

C:\LabSolutions\Data\Project1\Bénédicté\BSD 03 093 C1 - 10µ.l

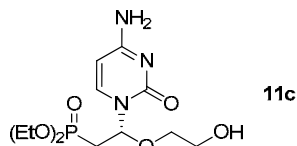
(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**



$C_{12}H_{22}N_3O_6P$
MW = 335.36 g.mol⁻¹
Colorless oil (35 mg, 37 %)



(±)-Diethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **11c**



$C_{12}H_{22}N_3O_6P$
MW = 335.36 g.mol⁻¹
Colorless oil (35 mg, 37 %)

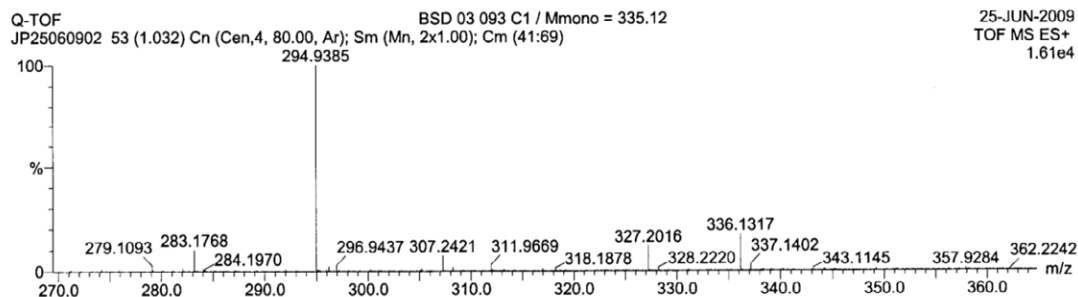
Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -10.0, max = 100.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

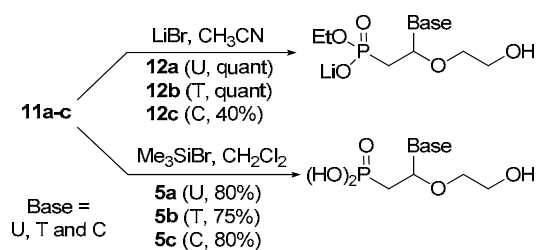
Monoisotopic Mass, Odd and Even Electron Ions
189 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)



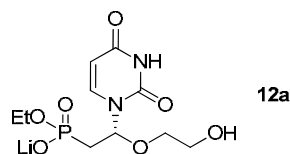
Minimum: -10.0
Maximum: 100.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
336.1317	336.1324	-0.7	-2.2	3.5	1	C12 H23 N3 O6 P ←
	336.1298	1.9	5.7	-1.0	2	C9 H25 N2 O9 P

II.12. (±)-Lithium ethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **12a**

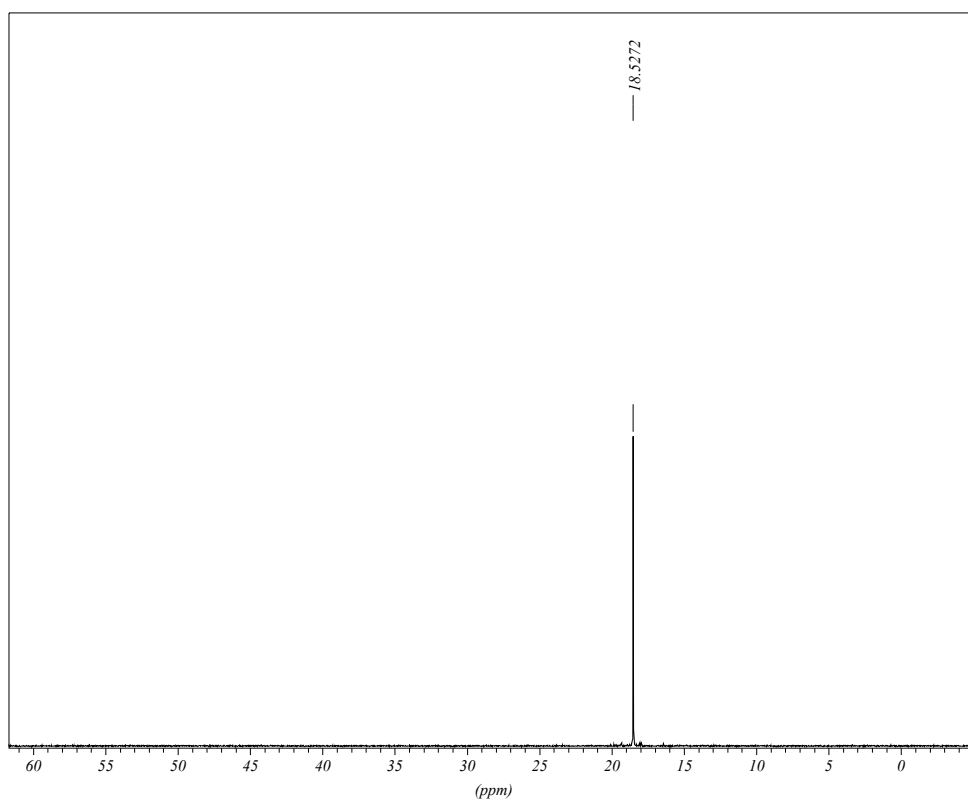


Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**



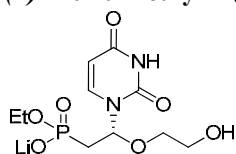
$\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_7\text{PLi}$
MW = 314.17 g.mol⁻¹
White solid (15 mg, 76%)

BSD 03 017P31CPD D2O opt/topspin cristau 38



*** Current Data Parameters ***
NAME : brut
EXPNO : 210
PROCNO : 0
*** Acquisition Parameters ***
DATE_t : 05:13:41
DATE_d : Mar 23 2009
NS : 16
OI : 8098.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 20642.5000000
SFO1 : 161.9836718 MHz
SOLVENT : D2O
SW : 200.4371 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 61.70 ppm
Stop : -5.34 ppm
SR : 0.01 Hz
SOLVENT : ?

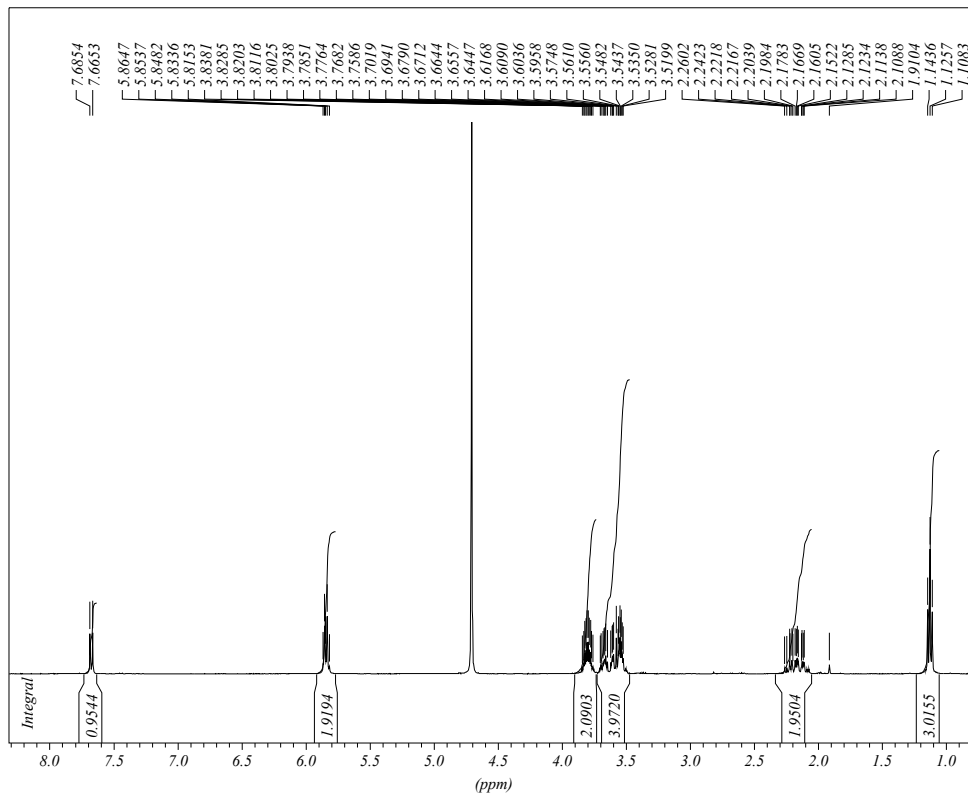
(±)-Lithium ethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **12a**



12a

$C_{10}H_{16}N_2O_7PLi$
MW = 314.17 g.mol⁻¹
White solid (15 mg, 76%)

BSD 03 017PROTON D2O opt/topspin cristau 38



*** Current Data Parameters ***

NAME : brut
EXPNO : 211
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 05:17:20
DATE_d : Mar 23 2009
NS : 16
O1 : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 114.000000
SFO1 : 400.1324008 MHz
SOLVENT : D2O
SW : 14.9831 ppm

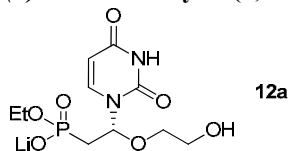
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

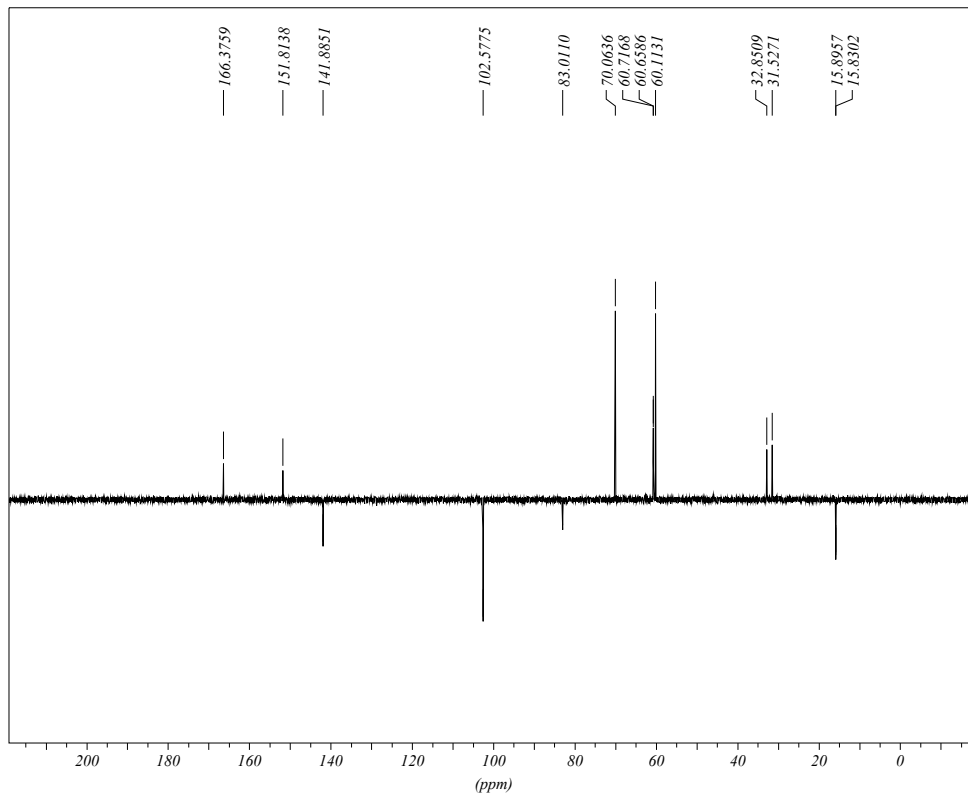
Start : 8.32 ppm
Stop : 0.75 ppm
SR : -0.00 Hz
SOLVENT : ?

(±)-Lithium ethyl 2-(2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **12a**



$C_{10}H_{16}N_2O_7PLi$
MW = 314.17 g.mol⁻¹
White solid (15 mg, 76%)

BSD 03 017C13APT D2O opt/topspin cristau 37



*** Current Data Parameters ***

NAME : brut
EXPNO : 212
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 11:05:55
DATE_d : Mar 24 2009
NS : 1024
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 26008.000000
SFO1 : 100.6228298 MHz
SOLVENT : D2O
SW : 238.3238 ppm

*** Processing Parameters ***

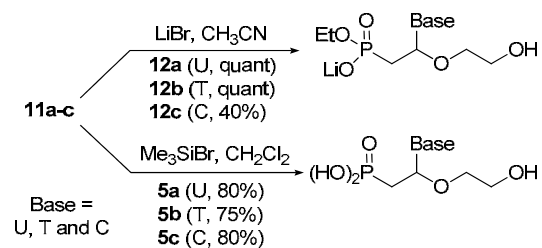
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

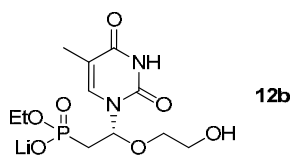
Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

II.13. (±)-Lithium ethyl hydroxyethoxy)ethylphosphonate 12b

2-(5-methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-

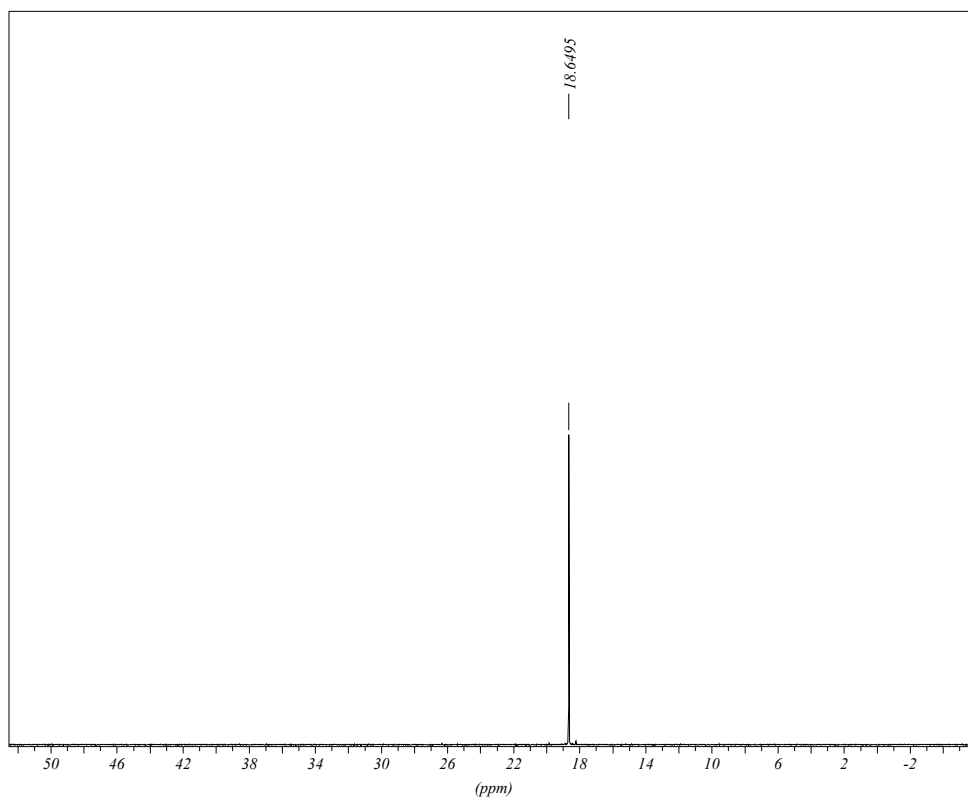


Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**

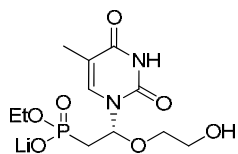


$\text{C}_{11}\text{H}_{18}\text{N}_2\text{O}_7\text{PLi}$
 MW = 328.19 g.mol⁻¹
 White solid (20 mg, 85%)

BSD 03 021P31CPD D2O opt/topspin cristau 57



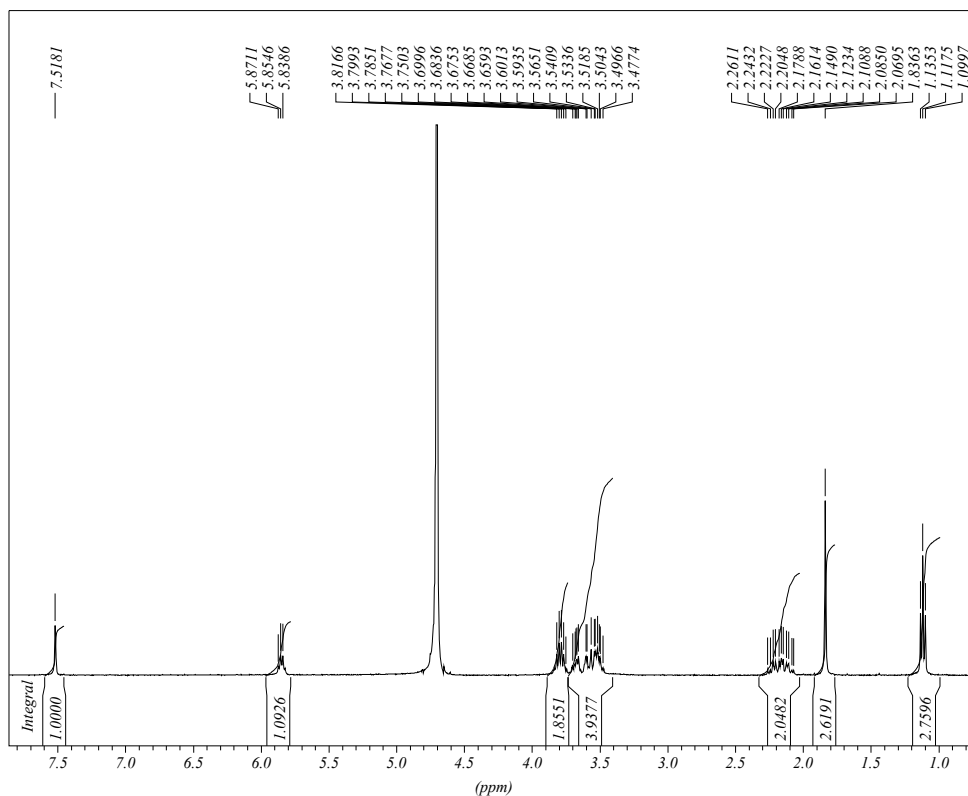
***** Current Data Parameters *****
 NAME : conc
 EXPNO : 210
 PROCNO : 0
***** Acquisition Parameters *****
 DATE_t : 05:33:49
 DATE_d : Apr 07 2009
 NS : 16
 O1 : 8098.78 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
 RG : 20642.5000000
 SFO1 : 161.9836718 MHz
 SOLVENT : D2O
 SW : 200.4371 ppm
***** Processing Parameters *****
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
***** 1D NMR Plot Parameters *****
 Start : 52.54 ppm
 Stop : -6.13 ppm
 SR : 0.01 Hz
 SOLVENT : ?



12b

$C_{11}H_{18}N_2O_7PLi$
MW = 328.19 g.mol⁻¹
White solid (20 mg, 85%)

BSD 03 021 PROTON D2O opt/topspin cristau 31



*** Current Data Parameters ***

NAME : 021
EXPNO : 211
PROCNO : 0

*** Acquisition Parameters ***

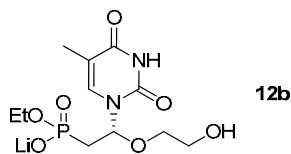
DATE_t : 04:05:44
DATE_d : Apr 01 2009
NS : 24
OI : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84
RG : 406.3999939
SFO1 : 400.1324008 MHz
SOLVENT : D2O
SW : 14.9831 ppm

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

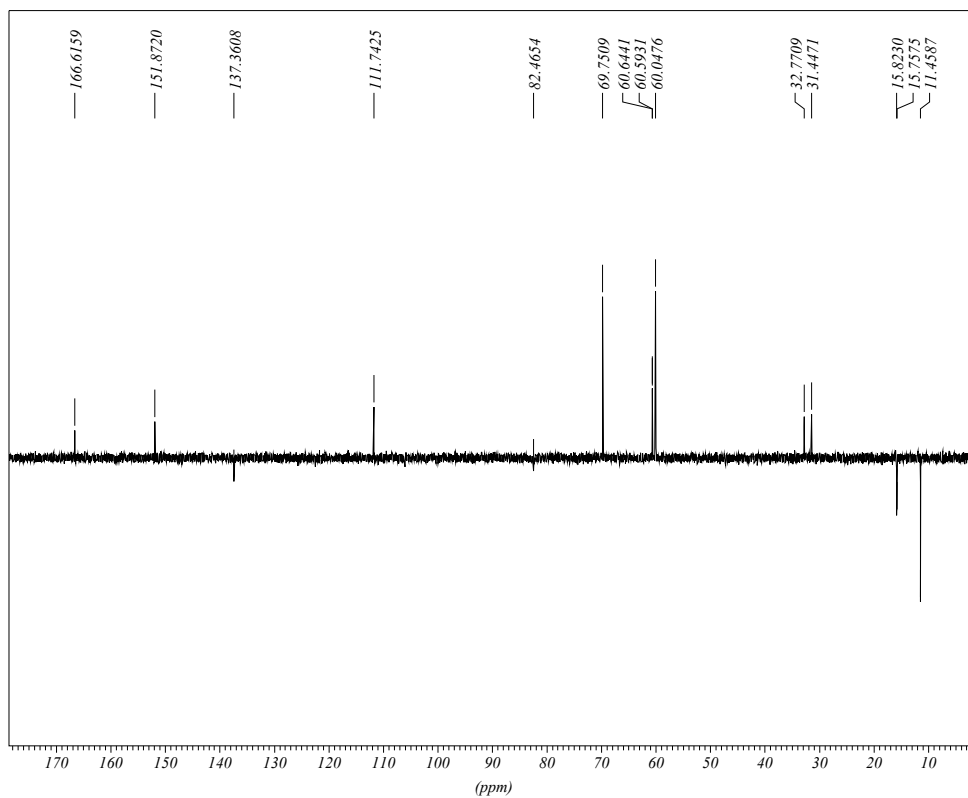
*** 1D NMR Plot Parameters ***

Start : 7.86 ppm
Stop : 0.71 ppm
SR : -0.00 Hz
SOLVENT : ?



$C_{11}H_{18}N_2O_7PLi$
MW = 328.19 g.mol⁻¹
White solid (20 mg, 85%)

BSD 03 021C13APT D2O opt/topspin cristau 57



*** Current Data Parameters ***

NAME : conc
EXPNO : 212
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 17:43:23
DATE_d : Apr 07 2009
NS : 256
O1 : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z84G
RG : 26008.000000
SFO1 : 100.6228298 MHz
SOLVENT : D2O
SW : 238.3238 ppm

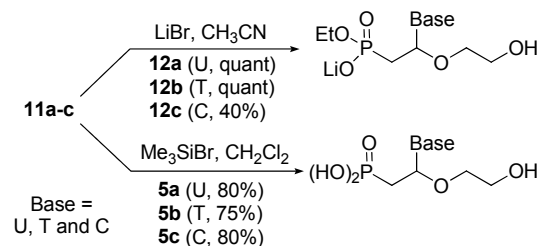
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

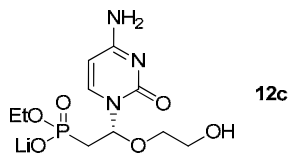
*** 1D NMR Plot Parameters ***

Start : 178.70 ppm
Stop : 0.85 ppm
SR : 0.00 Hz
SOLVENT : ?

II.14. (±)-Lithium ethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate 12c

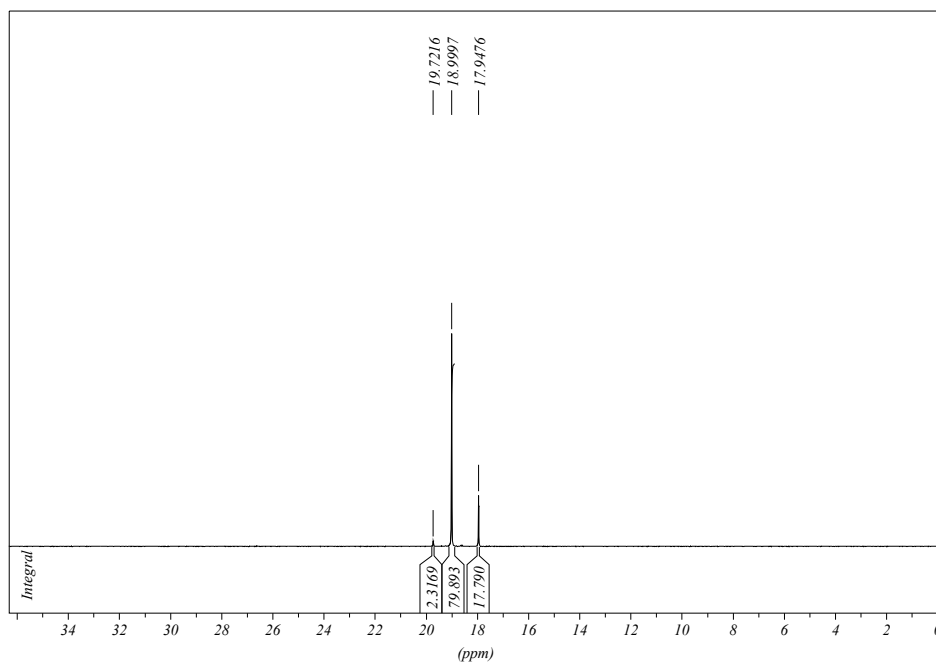


Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**



$\text{C}_{10}\text{H}_{17}\text{N}_3\text{O}_6\text{PLi}$
 MW = 309.36 g.mol⁻¹
 80% pure by phosphorus NMR

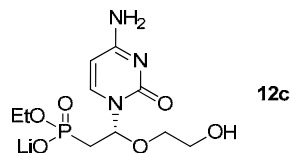
BSD03-121 / D2O



*** Current Data Parameters ***

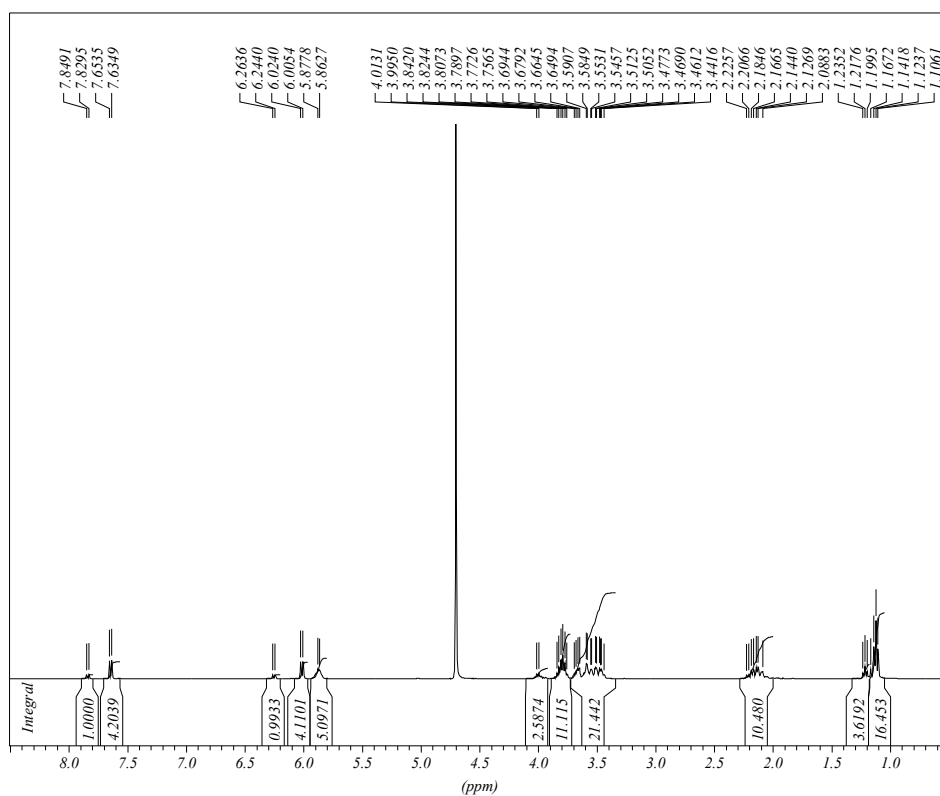
NAME : BSD03--1
 EXPNO : 1-PHOS-1
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 03:36:34
 DATE_d : Sep 11 2009
 NS : 64
 OI : 12958.05 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-GRD Z
 RG : 6502.000000
 SFO1 : 161.9885510 MHz
 SOLVENT : D2O
 SW : 400.8621 ppm
 *** Processing Parameters ***
 GB : 0.000000
 LB : 1.00 Hz
 SI : 32768
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 36.31 ppm
 Stop : -0.20 ppm
 SR : -0.01 Hz
 SOLVENT : ?

(±)-Lithium ethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **12c**



C₁₀H₁₇N₃O₆PLi
MW = 309.36 g.mol⁻¹
80% pure by phosphorus NMR

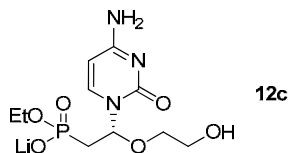
BSD03-121/D20



*** Current Data Parameters ***

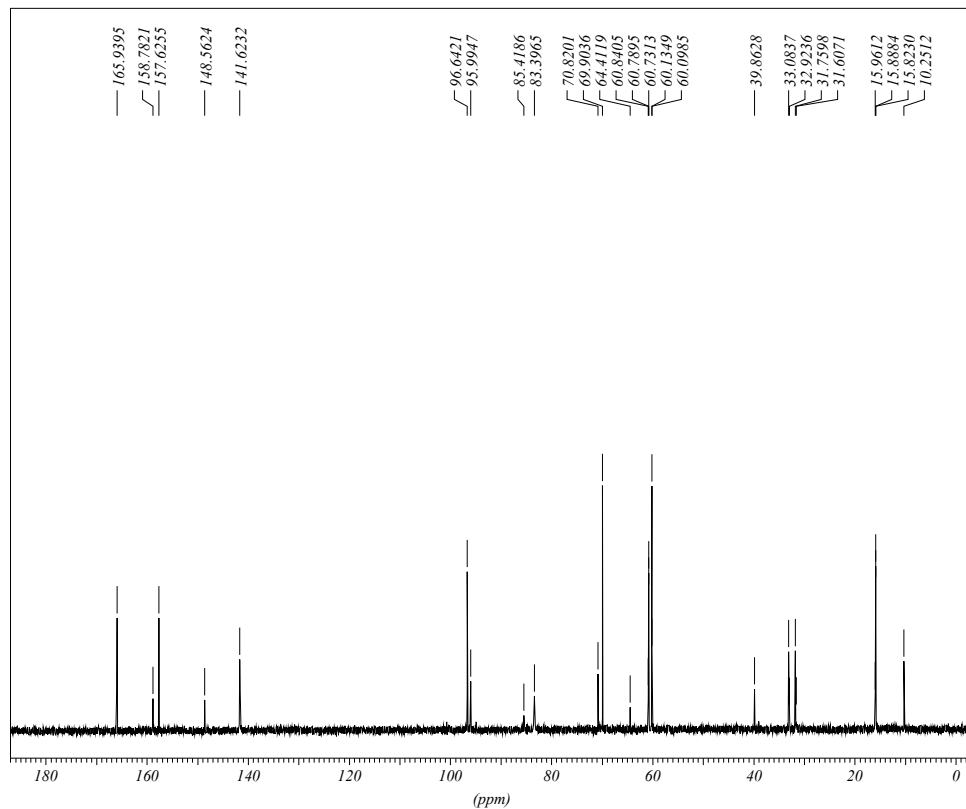
NAME : BSD03--1
EXPNO : 20-PRO-1
PROCNO : 0
*** Acquisition Parameters ***
DATE_1 : 17:29:20
DATE_d : Sep 10 2009
NS : 64
OI : 3000.97 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-GRD Z
RG : 161.3000031
SFO1 : 400.1330010 MHz
SOLVENT : D2O
SW : 16.0203 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 0.10 Hz
SI : 32768
TI : CC159P/D2O
*** 1D NMR Plot Parameters ***
Start : 8.51 ppm
Stop : 0.49 ppm
SR : -0.00 Hz
SOLVENT : ?

(±)-Lithium ethyl 2-(4-amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonate **12c**



$C_{10}H_{17}N_3O_6PLi$
MW = 309.36 g.mol⁻¹
80% pure by phosphorus NMR

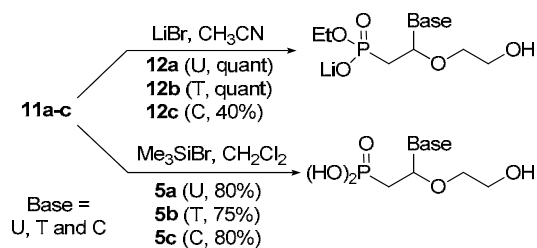
BSD03-121/D2O



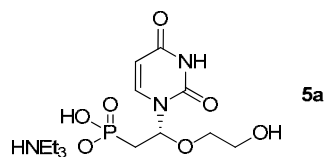
*** Current Data Parameters ***

NAME : BSD03--1
EXPNO : 21-PRO-1
PROCNO : 0
*** Acquisition Parameters ***
DATE_t : 23:03:30
DATE_d : Sep 10 2009
NS : 8192
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-GRD Z
RG : 5160.6000977
SFO1 : 100.6228298 MHz
SOLVENT : D2O
SW : 238.3238 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI : BO117/DMSOD6
*** 1D NMR Plot Parameters ***
Start : 187.04 ppm
Stop : -3.31 ppm
SR : 0.00 Hz
SOLVENT : ?

II.15. (±)-2-(2,4-Dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid triethyl ammonium salt **5a**

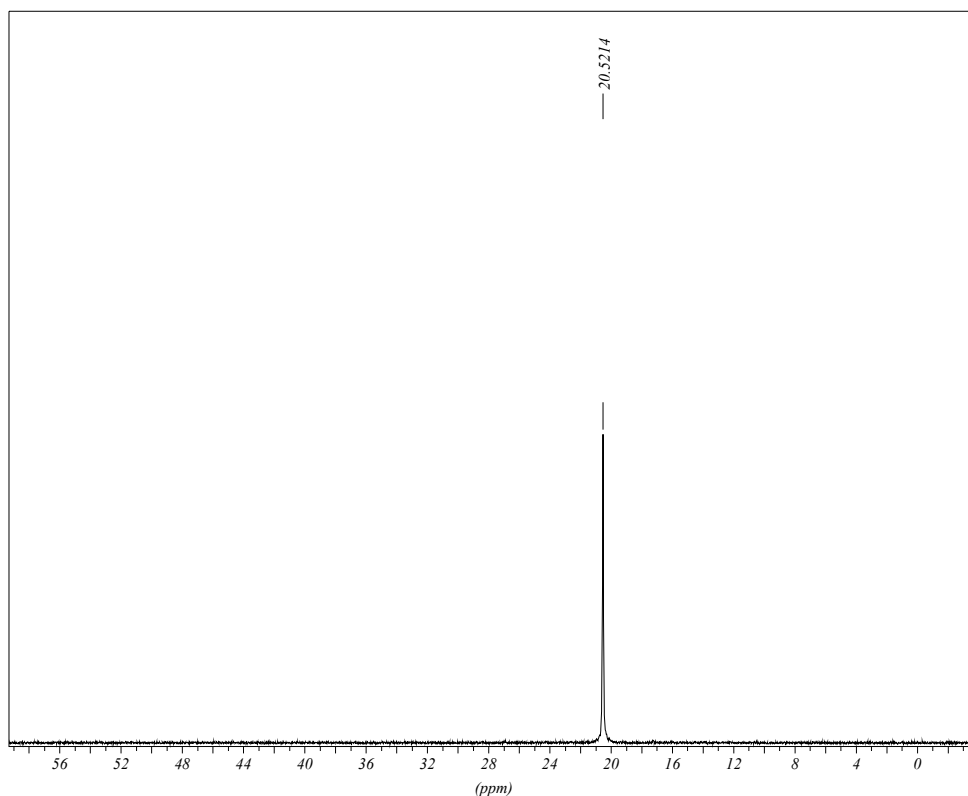


Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**



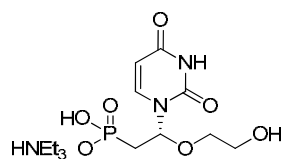
$\text{C}_8\text{H}_{13}\text{N}_2\text{O}_7\text{P}$ phosphonic diacid form
 $\text{C}_{14}\text{H}_{27}\text{N}_3\text{O}_7\text{P}$ triethylammonium salt
 $\text{MW}_{\text{diacid form}} = 280.18 \text{ g}\cdot\text{mol}^{-1}$
 $\text{MW}_{\text{ammonium salt}} = 380.37 \text{ g}\cdot\text{mol}^{-1}$
 Ammonium salt is a colorless oil (60 mg, 32 %)

BSD03 045 C18 F1P31CPD D2O opt/topspin cristau 59



***** Current Data Parameters *****
 NAME : F1
 EXPNO : 203
 PROCNO : 0
***** Acquisition Parameters *****
 DATE_1 : 00:32:02
 DATE_2 : May 11 2009
 NS : 16
 O1 : 8098.78 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
 RG : 11585.2001953
 SFO1 : 161.9836718 MHz
 SOLVENT : D2O
 SW : 200.4371 ppm
***** Processing Parameters *****
 GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :
***** 1D NMR Plot Parameters *****
 Start : 59.33 ppm
 Stop : -3.98 ppm
 SR : 0.01 Hz
 SOLVENT : ?

(±)-2-(2,4-Dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid
triethylammonium salt **5a**



C₈H₁₃N₂O₇P phosphonic diacid form

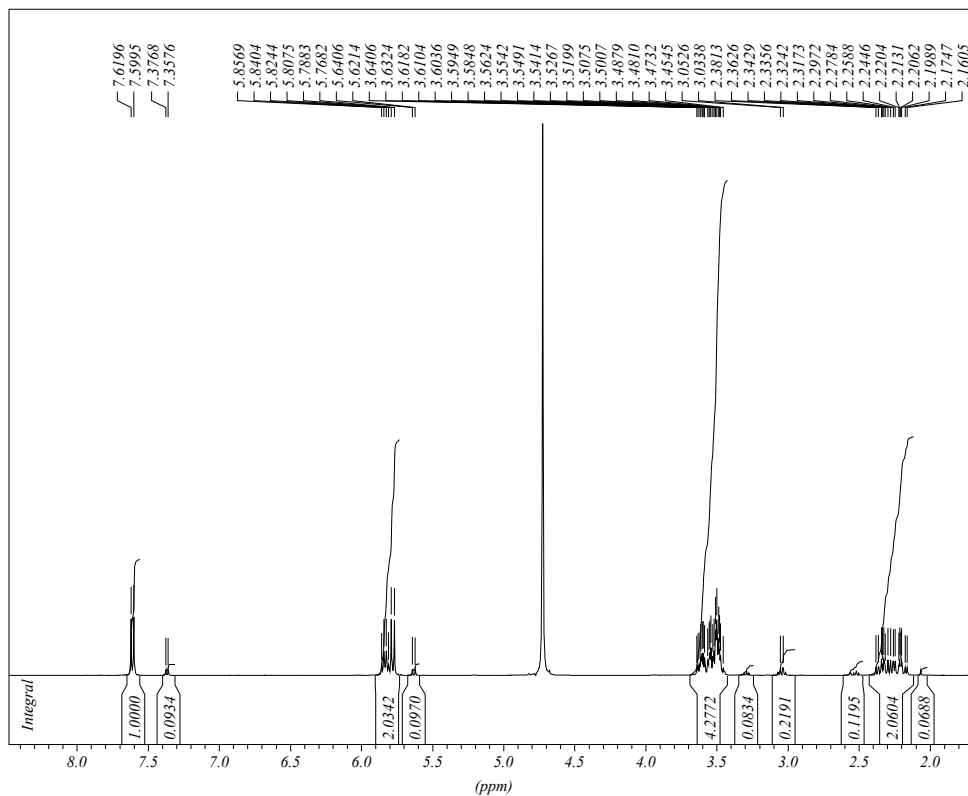
C₁₄H₂₇N₃O₇P triethylammonium salt

MW_{diacid form} = 280.18 g.mol⁻¹

MW_{ammonium salt} = 380.37 g.mol⁻¹

Ammonium salt is a colorless oil (60 mg, 32 %)

BSD 03 045 C18 F1PROTON D2O opt/topspin cristau 59



*** Current Data Parameters ***

NAME : F1

EXPNO : 204

PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 00:35:27

DATE_d : May 11 2009

NS : 16

O1 : 2400.78 Hz

PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846

RG : 64.0000000

SFO1 : 400.1324008 MHz

SOLVENT : D2O

SW : 14.9831 ppm

*** Processing Parameters ***

GB : 0.0000000

LB : 1.00 Hz

SI : 32768

TI :

*** 1D NMR Plot Parameters ***

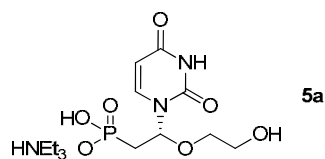
Start : 8.48 ppm

Stop : 1.66 ppm

SR : -0.00 Hz

SOLVENT : ?

(±)-2-(2,4-Dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid
triethylammonium salt **5a**



C₈H₁₃N₂O₇P phosphonic diacid form

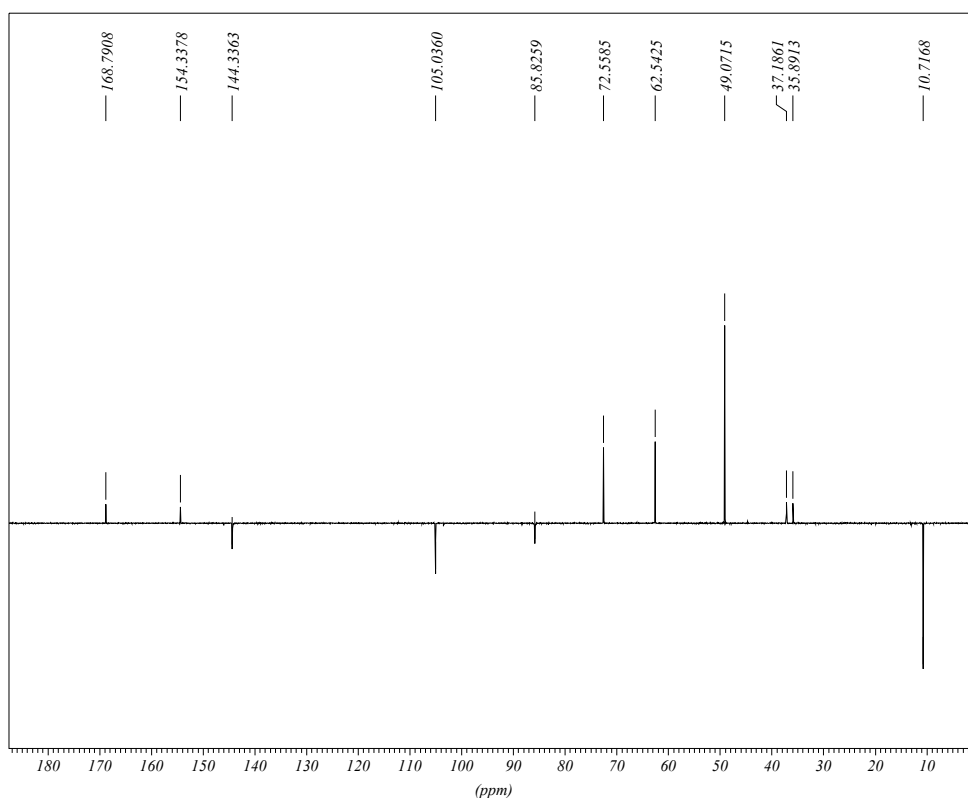
C₁₄H₂₇N₃O₇P triethylammonium salt

MW_{diacid form} = 280.18 g.mol⁻¹

MW_{ammonium salt} = 380.37 g.mol⁻¹

Ammonium salt is a colorless oil (60 mg, 32 %)

BSD 03 045 NEt3Cl3APT CDC13 opt/topspin cristau 22



*** Current Data Parameters ***

NAME : sech
EXPNO : 225
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 21:49:20
DATE_d : Jun 18 2009
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z846
RG : 26008.000000
SFO1 : 100.628298 MHz
SOLVENT : CDC13
SW : 238.3238 ppm

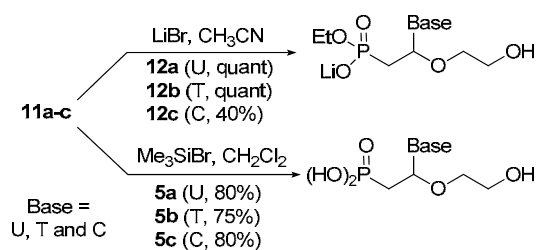
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

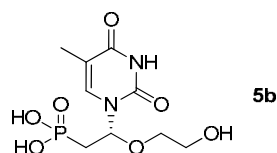
*** 1D NMR Plot Parameters ***

Start : 187.58 ppm
Stop : 0.05 ppm
SR : 0.00 Hz
SOLVENT : ?

II.16. (±)-2-(5-Methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5b**

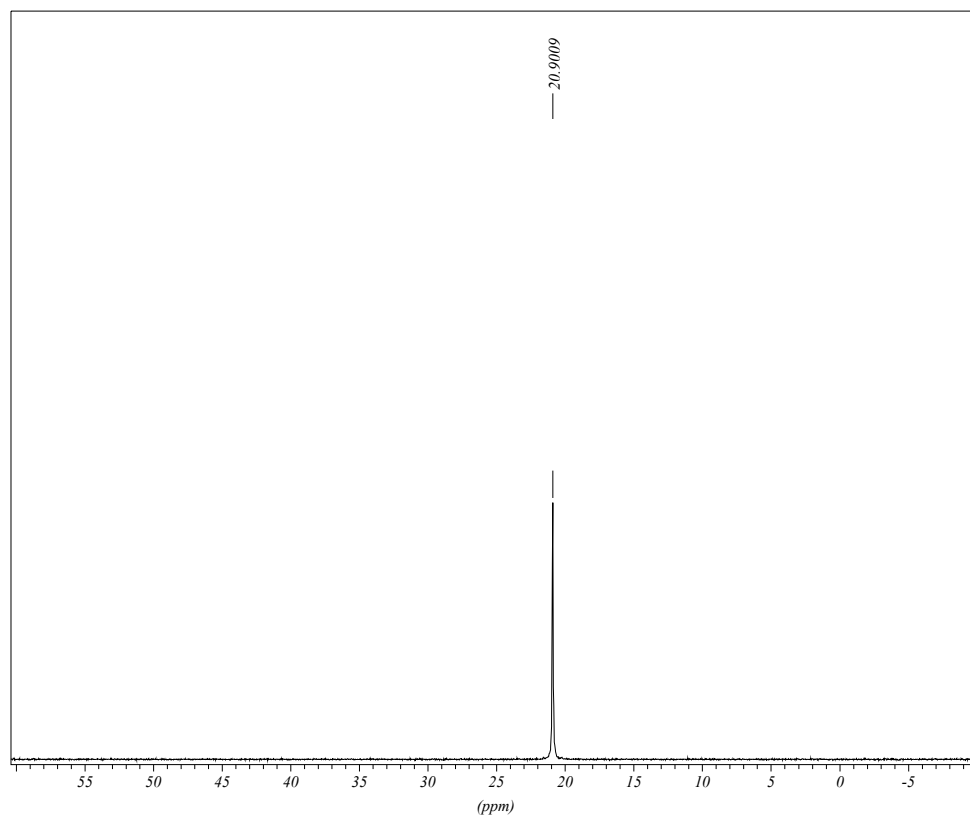


Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**



$C_9H_{15}N_2O_7P$
 $MM = 294.20 \text{ g.mol}^{-1}$
 White solid (116 mg, 74%)

BSD 03 097



*** Current Data Parameters ***

NAME : sech
 EXPNO : 201
 PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 09:35:11
 DATE_d : Jul 16 2009
 NS : 32
 O1 : -5062.76 Hz
 PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD 2
 RG : 11585.2001953
 SFO1 : 101.2494172 MHz

SOLVENT : D2O
 SW : 401.4878 ppm

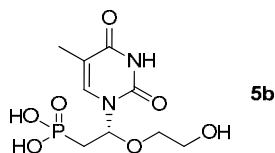
*** Processing Parameters ***

GB : 0.0000000
 LB : 1.00 Hz
 SI : 32768
 TI :

*** 1D NMR Plot Parameters ***

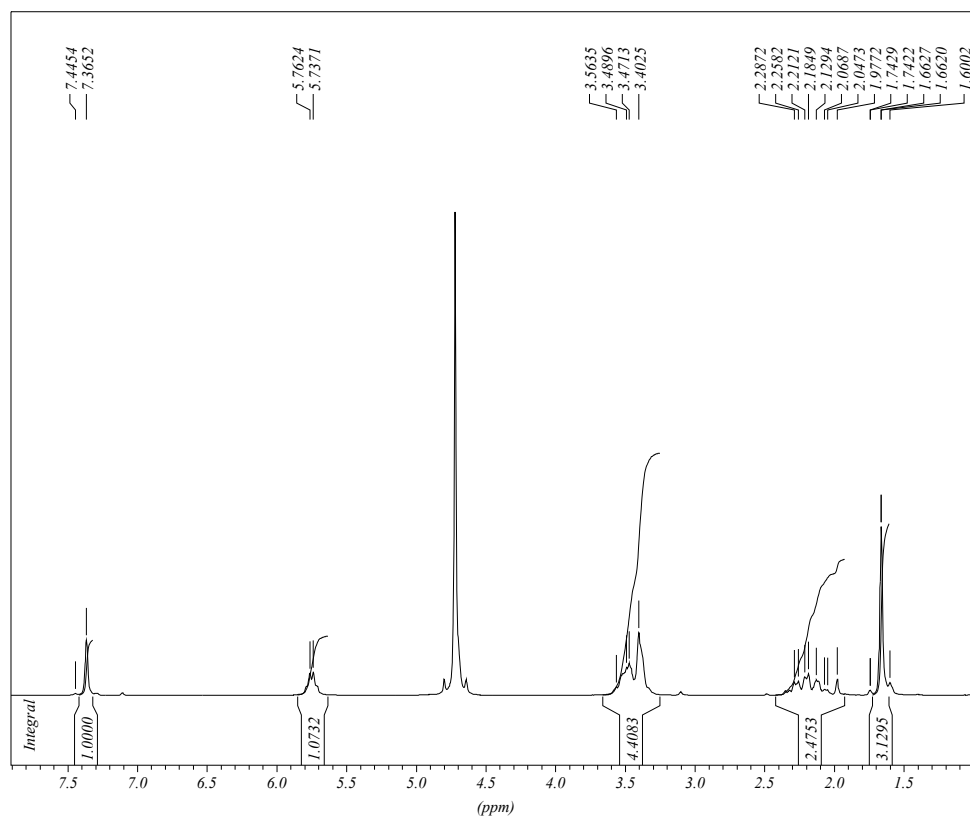
Start : 60.39 ppm
 Stop : -10.26 ppm
 SR : 0.00 Hz
 SOLVENT : ?

(±)-2-(5-Methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5b**



C₉H₁₅N₂O₇P
MM = 294.20 g.mol⁻¹
White solid (116 mg, 74%)

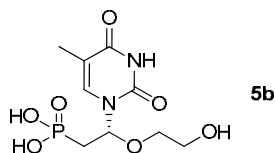
BSD 03 097



*** Current Data Parameters ***

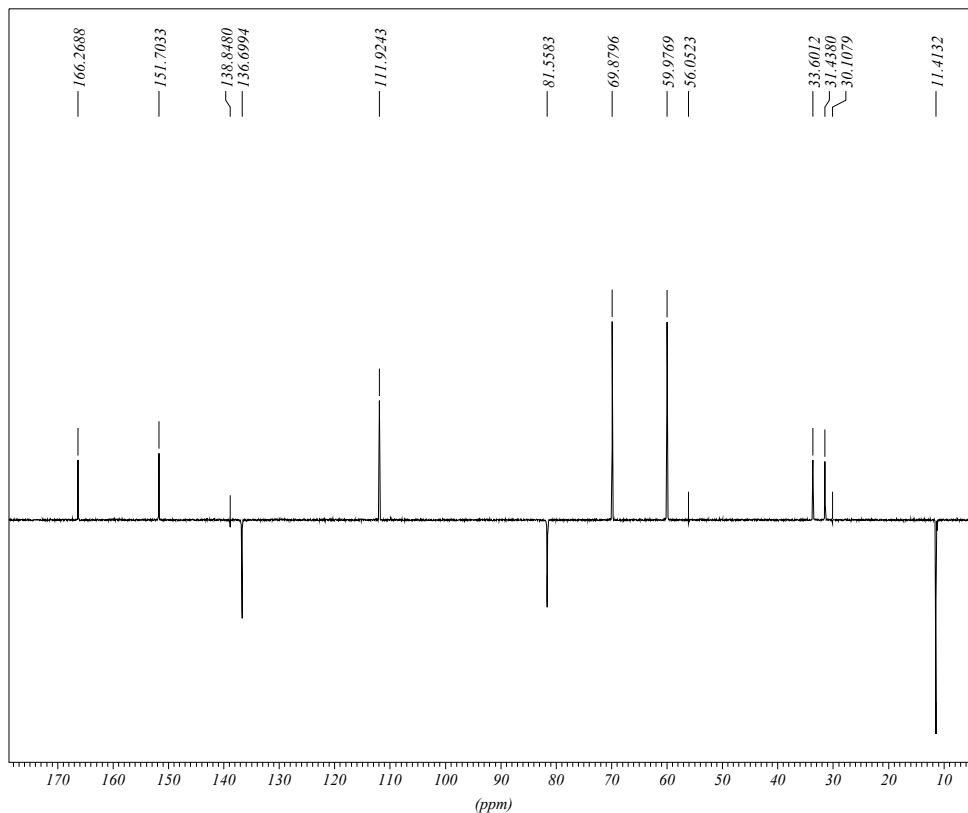
NAME : sech
EXPNO : 202
PROCNO : 0
*** Acquisition Parameters ***
DATE_t : 09:37:46
DATE_d : Jul 16 2009
NS : 32
O1 : 1544.66 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
RG : 90.5000000
SFO1 : 250.1315447 MHz
SOLVENT : D2O
SW : 20.6930 ppm
*** Processing Parameters ***
GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 7.91 ppm
Stop : 0.95 ppm
SR : -0.00 Hz
SOLVENT : ?

(±)-2-(5-Methyl-2,4-dioxo-3,4-dihydro-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5b**



$C_9H_{15}N_2O_7P$
MM = 294.20 g.mol⁻¹
White solid (116 mg, 74%)

BSD03097



*** Current Data Parameters ***

NAME : sech
EXPNO : 210
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 15:26:50
DATE_d : Jul 16 2009
NS : 2048
O1 : 6289.05 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
RG : 16384.0000000
SFO1 : 62.9015280 MHz
SOLVENT : D2O
SW : 239.4257 ppm

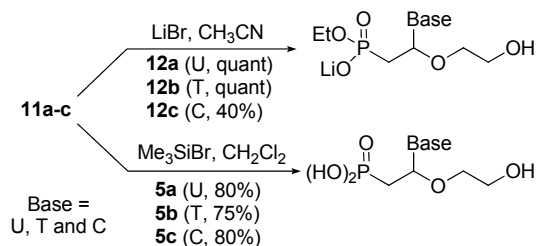
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

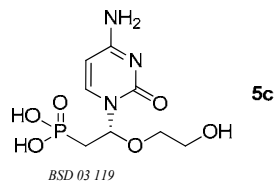
*** 1D NMR Plot Parameters ***

Start : 178.82 ppm
Stop : 3.77 ppm
SR : 0.00 Hz
SOLVENT : ?

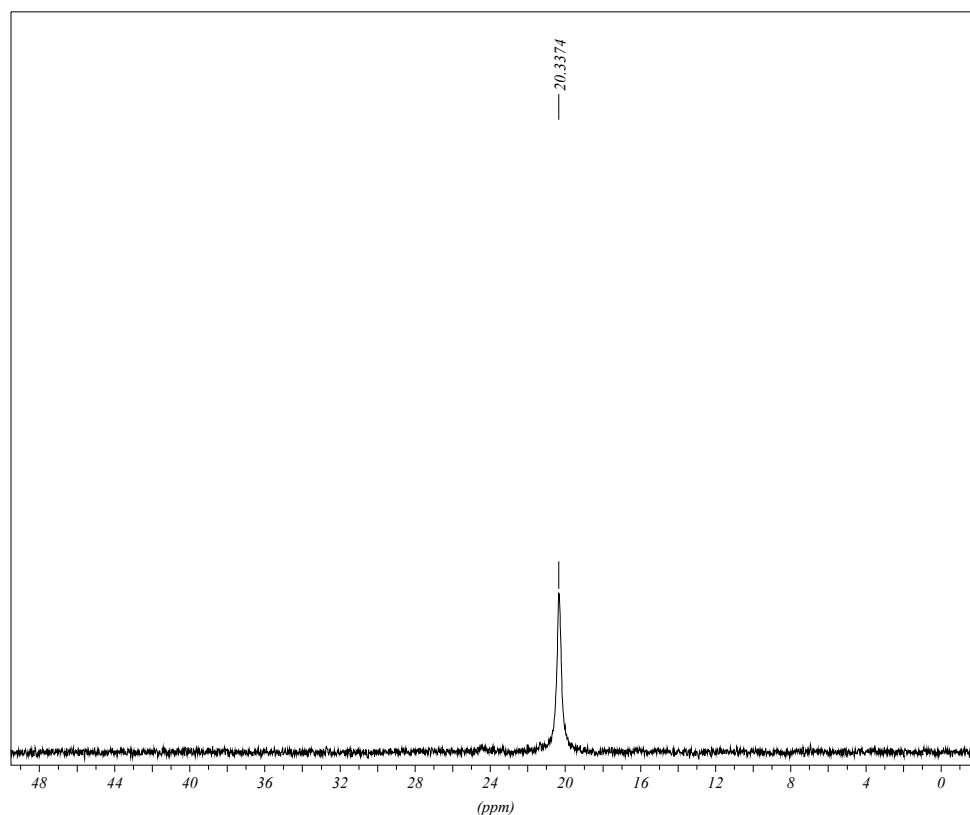
II.17. (±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5c**



Scheme 4 Synthesis of free phosphonic acid and mono-lithium salt derivatives **5a-c** and **12a-c**

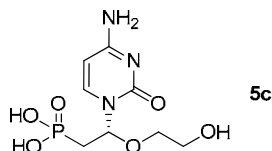


$\text{C}_8\text{H}_{14}\text{N}_3\text{O}_6\text{P}$
MW = 279.19 g.mol⁻¹
Colorless oil (81 mg, 97%)



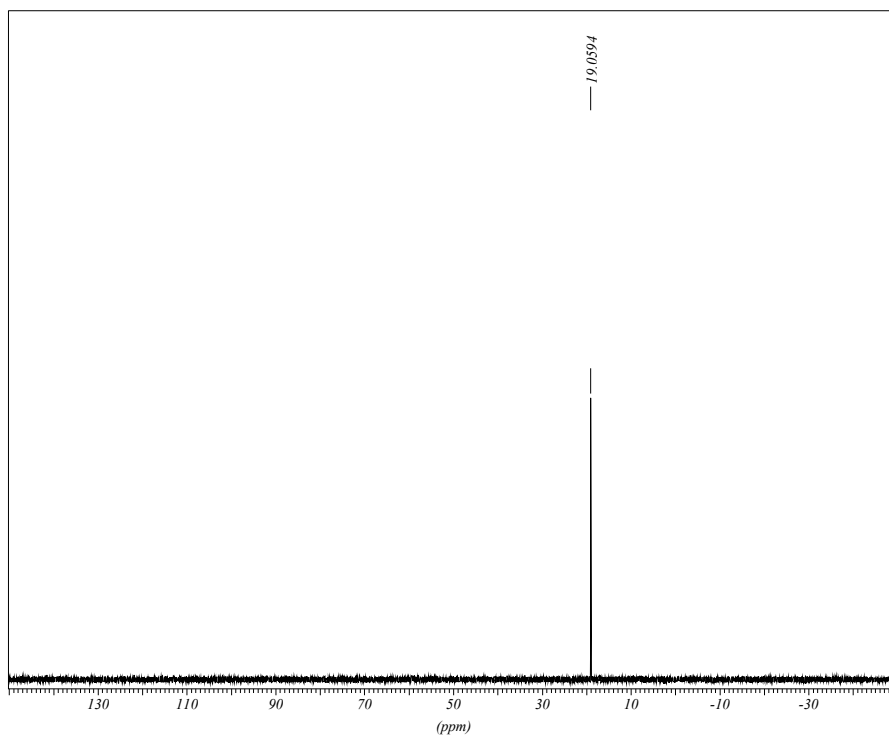
*** Current Data Parameters ***
NAME : 119
EXPNO : 202
PROCNO : 0
*** Acquisition Parameters ***
DATE_t : 01:49:45
DATE_d : Jul 17 2009
NS : 16
OI : -5062.76 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
RG : 8192.000000
SFO1 : 101.2494172 MHz
SOLVENT : D2O
SW : 401.4878 ppm
*** Processing Parameters ***
GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :
*** 1D NMR Plot Parameters ***
Start : 49.52 ppm
Stop : -2.11 ppm
SR : 0.00 Hz
SOLVENT : ?

(±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5c**



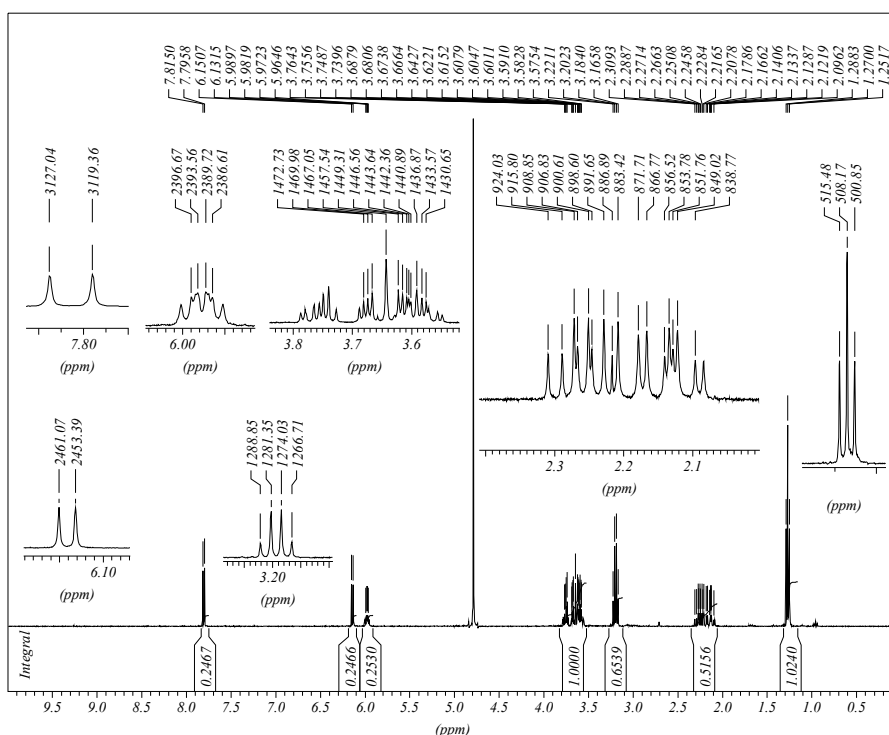
$C_8H_{14}N_3O_6P$
 MW = 279.19 g.mol⁻¹
 Colorless oil (81 mg, 97%)
 Triethylammonium salt

BSD 04 069 FIP31CPD CDCl3 opt/topspin cristau 58



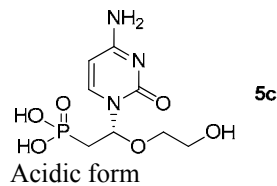
*** Current Data Parameters ***
 NAME : i9
 EXPNO : 210
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 08:29:58
 DATE_d : Jan 18 2010
 DE : 8.0 usec
 DS : 4
 NS : 16
 NUC1 : 31P
 O1 : 8098.78 Hz
 O2 : 1600.52 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840
 RG : 20642.5000000
 SFO1 : 161.9836718 MHz
 SW : 200.4371 ppm
 SW_h : 32467.532 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 1.00 Hz
 PC : 1.40
 SF : 161.975730 MHz
 *** 1D NMR Plot Parameters ***
 Start : 150.22 ppm
 Stop : -50.22 ppm
 SR : 0.01 Hz
 ppm_cm : 9.50
 Hz_cm : 1538.75

BSD 04 069 FIPROTON CDCl3 opt/topspin cristau 58



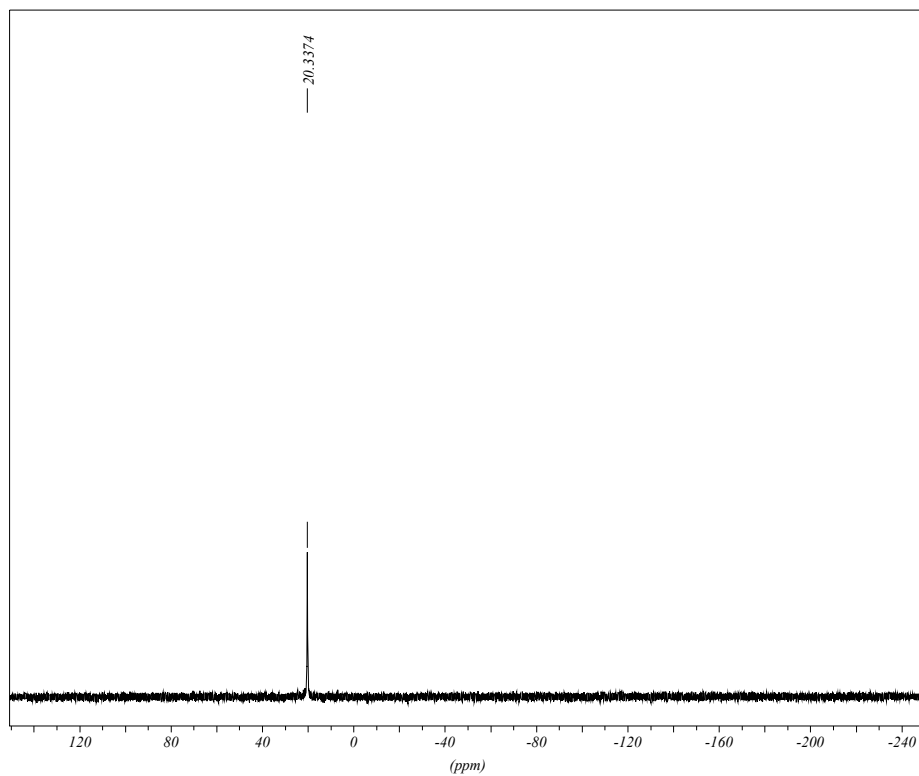
*** Current Data Parameters ***
 NAME : i9
 EXPNO : 211
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 08:33:27
 DATE_d : Jan 18 2010
 DE : 8.0 usec
 DS : 2
 NS : 16
 NUC1 : 1H
 O1 : 2400.78 Hz
 O2 : 2470.97 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD Z840
 RG : 406.3999939
 SFO1 : 400.1324008 MHz
 SW : 14.9831 ppm
 SW_h : 5995.204 Hz
 TD : 65536
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 0.10 Hz
 PC : 1.00
 SF : 400.1309810 MHz
 *** 1D NMR Plot Parameters ***
 Start : 10.00 ppm
 Stop : 0.04 ppm
 SR : 981.48 Hz
 ppm_cm : 0.47
 Hz_cm : 188.87

(±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5c**



$C_8H_{14}N_3O_6P$
MW = 279.19 g.mol⁻¹
Colorless oil (81 mg, 97%)

BSD 03 119



*** Current Data Parameters ***

NAME : 119
EXPNO : 202
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 01:49:45
DATE_d : Jul 17 2009
DE : 10.0 usec
DS : 4
NS : 16
NUC1 : 31P
O1 : -5062.76 Hz
O2 : 1000.52 Hz

PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD 2

RG : 8192.000000
SFO1 : 101.2494172 MHz
SW : 401.4878 ppm
SW_h : 40650.407 Hz
TD : 65536

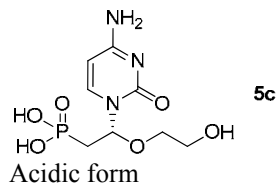
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 101.2544800 MHz

*** 1D NMR Plot Parameters ***

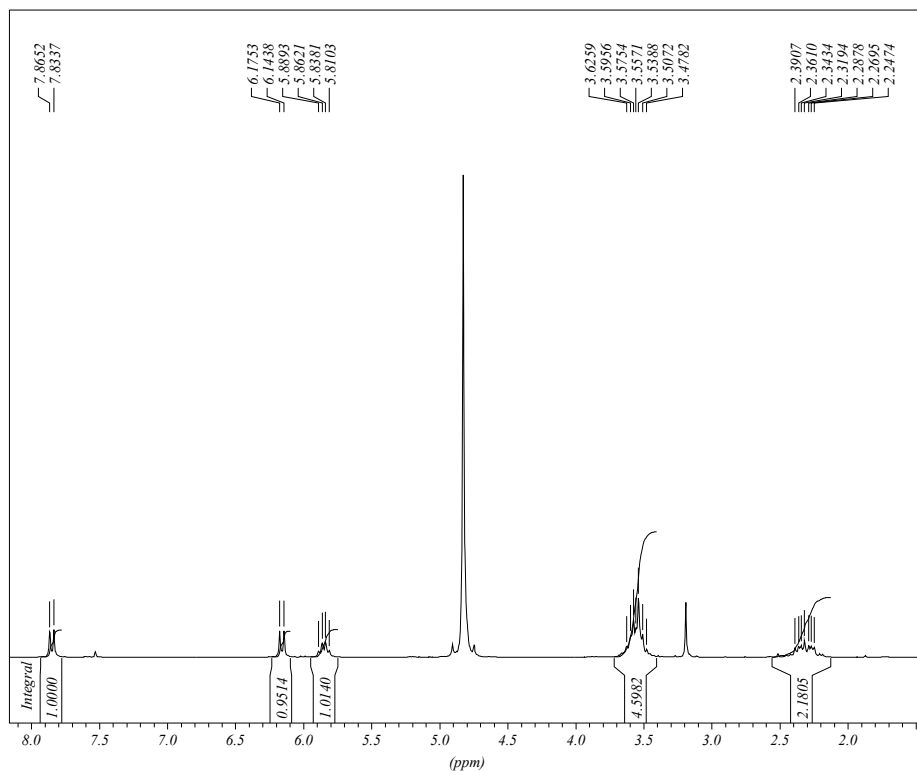
Start : 150.73 ppm
Stop : -250.73 ppm
SR : 0.00 Hz
ppm_cm : 19.03
Hz_cm : 1926.56

(±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5c**



BSD 03 119

$C_8H_{14}N_3O_6P$
MW = 279.19 g.mol⁻¹
Colorless oil (81 mg, 97%)



*** Current Data Parameters ***

NAME : 119
EXPNO : 201
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 01:40:27
DATE_d : Jul 17 2009
NS : 16
OI : 1544.66 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD 2
RG : 114.000000
SFO1 : 250.1315447 MHz
SOLVENT : D2O
SW : 20.6930 ppm

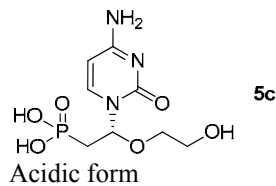
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

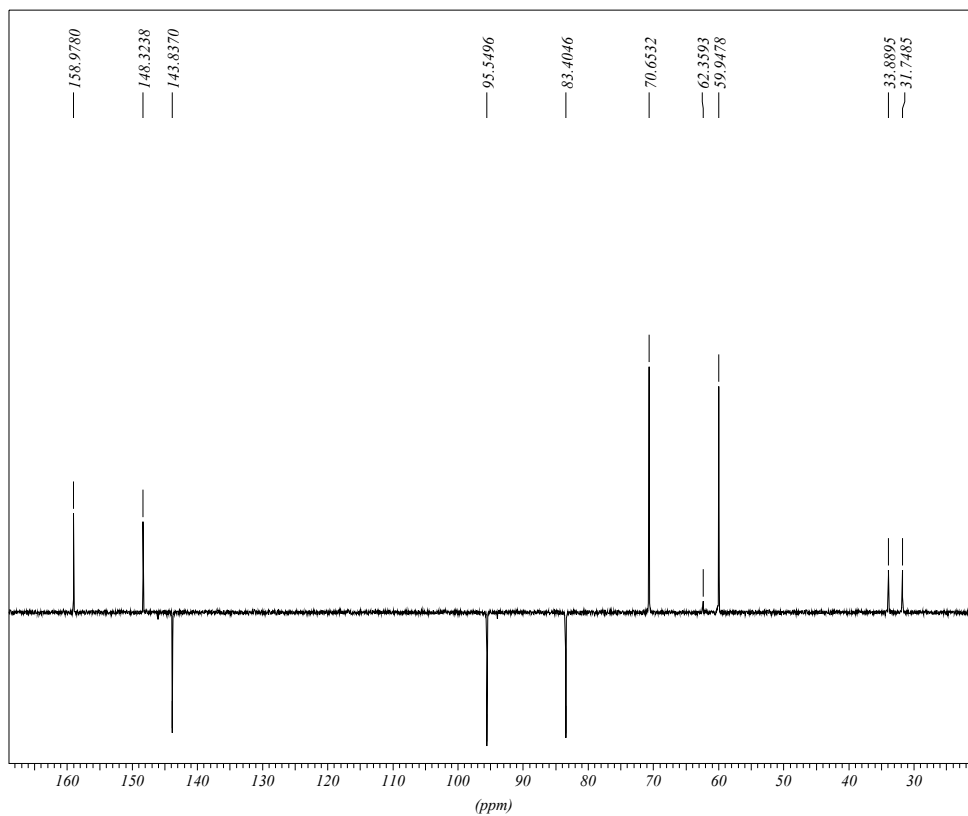
Start : 8.16 ppm
Stop : 1.43 ppm
SR : -25.57 Hz
SOLVENT : ?

(±)-2-(4-Amino-2-oxo-2H-pyrimidin-1-yl)-2-(2-hydroxyethoxy)ethylphosphonic acid **5c**



BSD 03 119 C1

$C_8H_{14}N_3O_6P$
MW = 279.19 g.mol⁻¹
Colorless oil (81 mg, 97%)



*** Current Data Parameters ***

NAME : C1CONT-1
EXPNO : 232
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 12:58:42
DATE_d : Nov 24 2009
NS : 2048
O1 : 6289.05 Hz
PROBHD : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
RG : 16384.000000
SFO1 : 62.9015280 MHz
SOLVENT : D2O
SW : 239.4257 ppm

*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
SI : 32768
TI :

*** 1D NMR Plot Parameters ***

Start : 168.94 ppm
Stop : 20.11 ppm
SR : -0.00 Hz
SOLVENT : ?